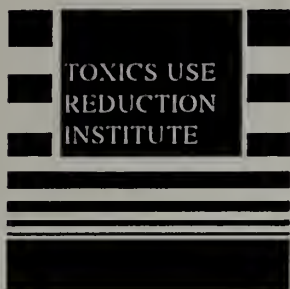


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**CYANIDE-BASED
ELECTROPLATING ELIMINATION**

NORTHROP GRUMMAN CORPORATION

Technical Report No. 20

1994

University of Massachusetts Lowell

CYANIDE-BASED ELECTROPLATING ELIMINATION

NORTHROP GRUMMAN CORPORATION ELECTRONICS SYSTEMS DIVISION - PRECISION PRODUCTS PLANT

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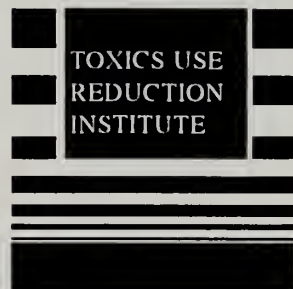
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December 1994

The Toxics Use Reduction Institute Matching Grants Program

The Toxics Use Reduction Institute
University of Massachusetts Lowell



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Toxics Use Reduction Institute Matching Grants Program

The Institute annually provides direct funding to Massachusetts industries on a matching basis for toxics use reduction (TUR) feasibility and technology studies. The matching Grants Program was initiated in FY 93 to facilitate the development and use of innovative techniques that reduce the use of toxic chemicals or the generation of toxic byproducts in Massachusetts businesses. Grants are awarded on a competitive basis for companies to conduct TUR studies at their facilities. Recipients prepare project reports which assist in transferring toxics use reduction technologies and methods to other companies.

Notice

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I. EXECUTIVE SUMMARY

Eliminating cyanide processes generates many benefits. The removal of extremely toxic chemicals from the workplace, and the reduction in waste treatment costs are among the most notable. This investigation demonstrates the replacement of copper cyanide based processes with a commercially available copper electroplating solution that contains no cyanides.

Alternative non-cyanide electroplating processes were evaluated for three substrates: aluminum, Kovar, and a nickel-iron alloy. A commercially available alkaline copper solution, E-Brite 30/30 (Electrochemical Products Inc.), was selected and evaluated. Test coupons and production parts were electroplated in the E-Brite 30/30 and subjected to a number of tests. Adhesion, solderability, visual appearances, and thicknesses of the coatings from the alternative process were evaluated with favorable results. Plating on zincated aluminum posed no difficulties with this bath although the plating rate was slower than with copper cyanide. The E-Brite 30/30 provided a drop-in alternative requiring no alteration in our current cleaning processes. However, it is important to note that this may not be true for all processes. The copper cyanide bath is much more tolerant of surface contamination than the E-Brite 30/30 bath.

Waste treatment of E-Brite 30/30 rinsewaters is limited to the metal ions, requiring only pH adjustment and precipitation. This eliminates the need for the separate cyanide destruction process required on the copper cyanide rinsewaters.

An economic evaluation of the E-Brite 30/30 process for zincated aluminum ribbon versus the copper cyanide process was performed. Significant indirect expense savings were realized. However, direct labor costs were higher. Overall, we projected total additional costs at \$626 per year for the non-cyanide process. In light of the substantial toxic use reduction benefits arising from eliminating the cyanide process, the small difference in projected cost between the two processes is inconsequential.

Therefore, based on our favorable analysis of this alternate copper electroplating process, we plan to pursue the conversion of our copper cyanide plating operations.

II. PROJECT SCOPE

This project demonstrates the use of non-cyanide based electroplating of copper on Aluminum, Kovar, and Nickel-Iron alloy parts. Northrop Grumman Corporation - Electronics

Systems Division - Precision Products Plant manufactures gyroscopes, accelerometers, and guidance systems employing these devices, for a variety of aerospace applications. Currently, two components experience cyanide-based copper electroplating. These electroplated components provide critical functionality for our products, thus dictating that any alternative process produce consistent and reliable results.

The project scope included determining the existence of suitable alternative processes, obtaining alternative solutions, and plating test coupons and production parts. Analytical testing of both the alternative plating solutions employed and the coupons and production parts was performed. The solution testing involved verifying or developing methods for routine plating bath analysis. The evaluation of the parts entailed visual appearance appraisals, thickness measurements, adhesion assessments, solderability testing, and examination by Scanning Electron Microscopy (SEM) for the integrity of the parts and the coating. An economic evaluation of the alternative process was also conducted.

III. ASSESSMENT & APPLICATION OF TECHNOLOGIES

A. Northrop Grumman Part Descriptions/Requirements

Our primary efforts centered on identifying a non-cyanide plating process for aluminum. We utilize a 6061 aluminum ribbon as an electrical current carrier and flex element within our products. The 6061 aluminum provides both the necessary corrosion resistance and mechanical properties required by our application. This ribbon, measuring 0.0005 inches thick by 0.012 inches wide, is an extremely delicate part. The copper plating of this ribbon permits its soldering to clips to form a flexlead assembly which is subsequently built into a gyroscope.

The current process entails a number of steps. First, the ribbon is wound onto a heat treat fixture on which it is initially heat treated and artificially aged. The heat treatment improves the material's mechanical properties and shapes the material into the desired coil configuration. The ribbon is then gently transferred to a plating fixture. Next, the surface is prepared following the general guidelines of ASTM B253-79, "Preparation of and Electroplating on Aluminum by the Zincate Process". A zincate immersion step followed by water rinsing is the final step prior to copper plating. The current zincate utilized is Alumon D, an Enthone product which is primarily a mixture of zinc oxide and sodium hydroxide. The copper cyanide bath in use is a standard tartrate type strike bath

containing both sodium and copper cyanide. After electroplating, approximately five turns of ribbon are cut off the fixture and soldered to two beryllium copper clips to form the flexlead assembly. Ten to twelve assemblies can be fabricated from one fixture. Finally, the remaining exposed copper plating is electrolytically stripped using a chromic acid solution. Figures 1 through 4 display the aluminum ribbon at various stages during its manufacturing cycle.

The development of the non-cyanide process dictated the deposition of a solderable coating, preferably copper, on a zincated aluminum surface. The deposit was required to be adherent, free from defects, readily solderable, and easily stripped without damage to the underlying substrate or components of the assembly. The alternative process could not alter the integrity of the thin aluminum ribbon, add additional cost to the product, or increase health and environmental risks.

The two remaining applications, the Kovar (Fe-Ni-Co), and the Alloy 52 (Fe-Ni) had only two criteria. Both of these materials are used in electrical feedthru applications and contain glass to metal seals. Therefore, the alternative plating process must maintain the integrity of the glass and its seals. Additionally, in both instances, the copper plating provides the basis for future tin/lead solder electroplating. Therefore, the replacement process must also produce good adhesion.

B. Potential Alternative Processes

In order to identify appropriate alternative processes a literature search was conducted for commercially available non-cyanide plating alternatives. Generally, the requirements for the aluminum electroplating were the prohibiting factors in this search. Discussions of the potential alternatives follow:

a. Electroless Copper Plating

Electroless copper solutions are primarily used within the printed circuit board industry for the plating of nonconductive material. They are usually alkaline in nature and require a catalyst for the deposition to occur. They produce highly stressed deposits creating brittle coatings. Since this aluminum ribbon is designed for its flexibility, a brittle coating would be unsuitable.

Figure 1 Aluminum Ribbon on Plating Fixture

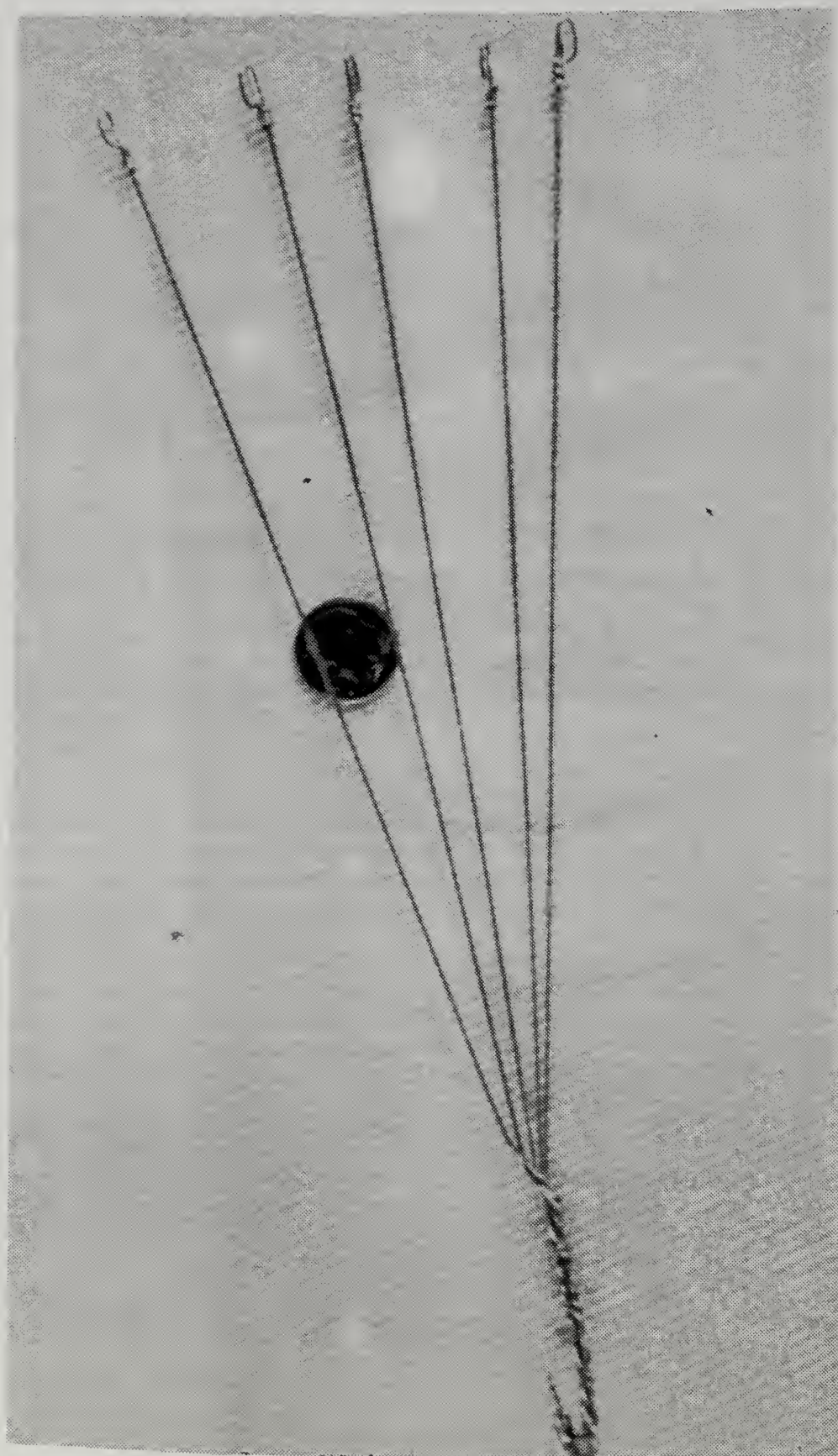


Figure 2 Plated Aluminum Ribbon on Plating Fixture



Figure 3 Final Flexlead Assembly After Copper Stripping Process

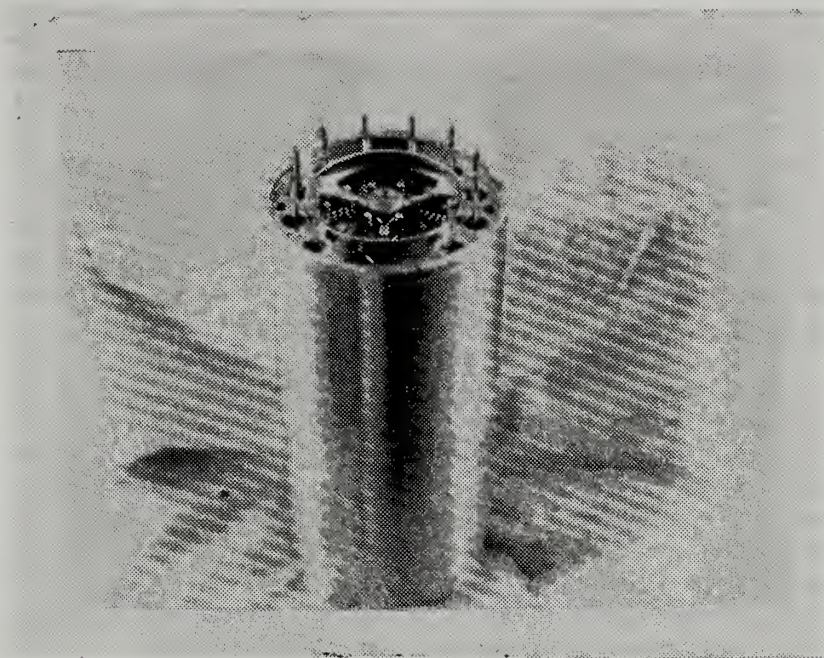


a. Approx.
Actual Size



b. 4.6X

Figure 4 Flexlead Assembly Installed in Gyroscope



a. 1.25X



b. 1.1X

b. Electroless Nickel Plating

Electroless nickel plating could work well on the feedthru applications but would be problematic with the aluminum ribbon. In fact, the Alloy 52 application is currently being procured as a completed feedthru with solder plating already applied on top of nickel. Unfortunately, our most stringent criteria lies with the aluminum ribbon which is soldered into an assembly. After soldering, the nickel would have to be stripped without attacking any portion of the assembly. At this time, no technique is readily available for stripping a nickel coating off of the flexlead assembly without radically altering the assembly itself.

c. Acid Copper Electroplating

Electrolytic acid copper solutions are typically employed to deposit thick coatings. They contain acids which would attack the aluminum substrate. Due to the thickness of the aluminum ribbon, any metal loss is unacceptable.

d. Alkaline Copper Electroplating

Electrolytic non-cyanide alkaline copper solutions displayed the most promise. Three potential commercial solutions for plating on zincated aluminum were identified; E-Brite 30/30 manufactured by Electrochemical Products (EPI), Inc. of New Berlin, WI, Cupral produced by Enthone-OMI of New Haven, CT, and HCP-200 from Chemtech Products (formerly Harstan) of St. Louis, MO.. Both EPI's and Enthone's technical data sheets are located in Appendix A.

We discovered that Chemtech had discontinued manufacturing their product. They ceased fabricating this product line due to lack of demand. However, they were willing to share the HCP-200's formulation with us as long as we agreed not to manufacture it commercially, or in large quantities. It is currently in use at Lawrence Livermore National Laboratory and discussed in an April, 1992 report entitled "Electroplating Waste Minimization at Lawrence Livermore National Laboratory". Nonetheless, we decided not to pursue this process at this time. There would have been minimal, if any, technical support for this product.

After telephone discussions with Enthone technical personnel, Cupral was also eliminated as a viable

candidate. This process requires complex bath analysis and an auxiliary purification cell. It is also sensitive to zinc contamination, therefore incompatible with our zincated aluminum process. We suspected that it could add considerable cost to our plating process.

The EPI product, E-Brite 30/30 remained as the sole contender. This product appeared to be relatively straight forward with no complicated bath analysis. Copper content, pH, and electrolyte concentration evaluations were the recommended analyses. Electrolyte concentration and copper content can be performed by titrations. An electronic pH meter is necessary for both the electrolyte titration and monitoring the pH. The bath is more sensitive to both organic and metallic contamination than the copper cyanide bath. Therefore, continuous carbon filtration is recommended for controlling the organics. Monitoring the inorganics by spectroscopy or alternate means is also recommended. Bath performance can be evaluated by Hull cell tests. This bath's waste treatment is limited to copper ions thus significantly reducing disposal costs compared to cyanide based process. As a result of our literature search, we decided to experiment with EPI's E-Brite 30/30.

IV. TEST OR IMPLEMENTATION RESULTS

A. Preliminary Evaluation of E-Brite 30/30

Coupon testing in the laboratory encompassed the initial assessment of the E-Brite 30/30 copper plating bath. Coupon plating began in a 500 milliliter pyrex beaker heated by a hot plate. A magnetic stirrer provided the bath agitation. A thermometer measured the temperature. Two oxygen free copper rod shaped anodes (approximately 3.1 inches long by 0.3 inches in diameter) transferred the current to the solution. The solution consisted of 50% E-Brite 30/30 and 50% of deionized (18 megohm) water. Although EPI's product literature allows the use of tap water, we utilized deionized water for both solution make up and some rinsing steps. A rectifier (0 to 10 volts, 0 to 10 amperes) connected in series with a multimeter supplied the current to the bath. The multimeter provided measurements of the actual current in the milliampere range. Three different coupon materials were utilized. In two cases, similar alloys were substituted due to lack of suitable in-house material for coupon testing. 2024 aluminum alloy was substituted for 6061 aluminum. Carpenter 49, another iron-nickel alloy, was employed in lieu of Alloy 52. Table 1 describes the coupon configurations.

Table 1 Coupon Types and Configurations

<u>Part</u> <u>Material</u>	<u>Coupon</u> <u>Material</u>	<u>Approx. Coupon</u> <u>Dimensions</u>	<u>Area to</u> <u>be Plated</u>
6061 Al	2024 Al	1" x 4" flat plate	.027 sq. ft.
Alloy 52	Carpenter 49	.5" dia. x .1" thick	.004 sq. ft.
Kovar	Kovar	.75" dia. x 1" thick	.008 sq. ft.

The preliminary coupon testing was performed in beakers to evaluate deposition rates and appropriate current densities. The coupons were cleaned in the same manner as the parts they represented. They were then immersed in the E-Brite 30/30 plating bath at $120^{\circ}\text{F} \pm 5^{\circ}\text{F}$ for 3 minutes \pm 5 seconds. The current density was varied and the resulting coating thicknesses measured. Figures 5 through 7 present the results. All of the coupons, regardless of material or current density, exhibited good adhesion. Most displayed acceptable surface appearances at 6 to 10X magnification except for the aluminum which experienced some burning at 40 amperes per square foot (ASF).

B. Secondary Coupon Testing

Next, utilizing our preliminary results, a minimum of 5 more coupons of each material were plated in both the copper cyanide production bath and the E-Brite 30/30 bath. For the E-Brite plating, both the Carpenter 49 and Kovar coupons were processed in the beaker setup. The aluminum coupons were performed in an 11 liter bath. All of the copper cyanide plating was performed in the 11 liter production bath.

The 11 liter baths are located in polypropylene tanks situated in a custom four tank Technic's Plating Module. This module is equipped with 3 (0-12 volt, 0-1 ampere DC) rectifiers. CAL 7000 temperature controllers regulate bath temperatures by controlling 500 watt Technic (Model HXL5110R11) heating elements. Automatic timers begin timing as soon as a current is applied within the plating bath. Serfilco chemical solution pumps, (Model APS3000A-STT) with capacities of 55 gallons per hour, circulate the baths. When in use, the E-Brite 30/30 solution pumps continuously through a 3 micron carbon pack filter to remove organic contaminants. A 15 micron polypropylene filter is used on the copper cyanide bath. Both baths utilize oxygen free copper (OFC) anodes which are rod shaped measuring 6 inches

Figure 5 Carpenter 49 - Preliminary Coupon Testing in E-Brite 30/30 Bath

E-Brite 30/30 Bath

Bath Temp = 120 F \pm 10 F

Bath Time = 3 min

Sample #	Current Density ASF	Current m/a	Adhesion	Visual Appearance	Thickness uin	Estimated Plating Rate uin/min
1	15	60	pass	ok	16.1	5.4
2	20	80	pass	ok	23.5	7.8
3	25	100	pass	ok	31.0	10.3
4	30	120	pass	ok	35.0	11.7
5	40	160	pass	ok	58.4	19.5

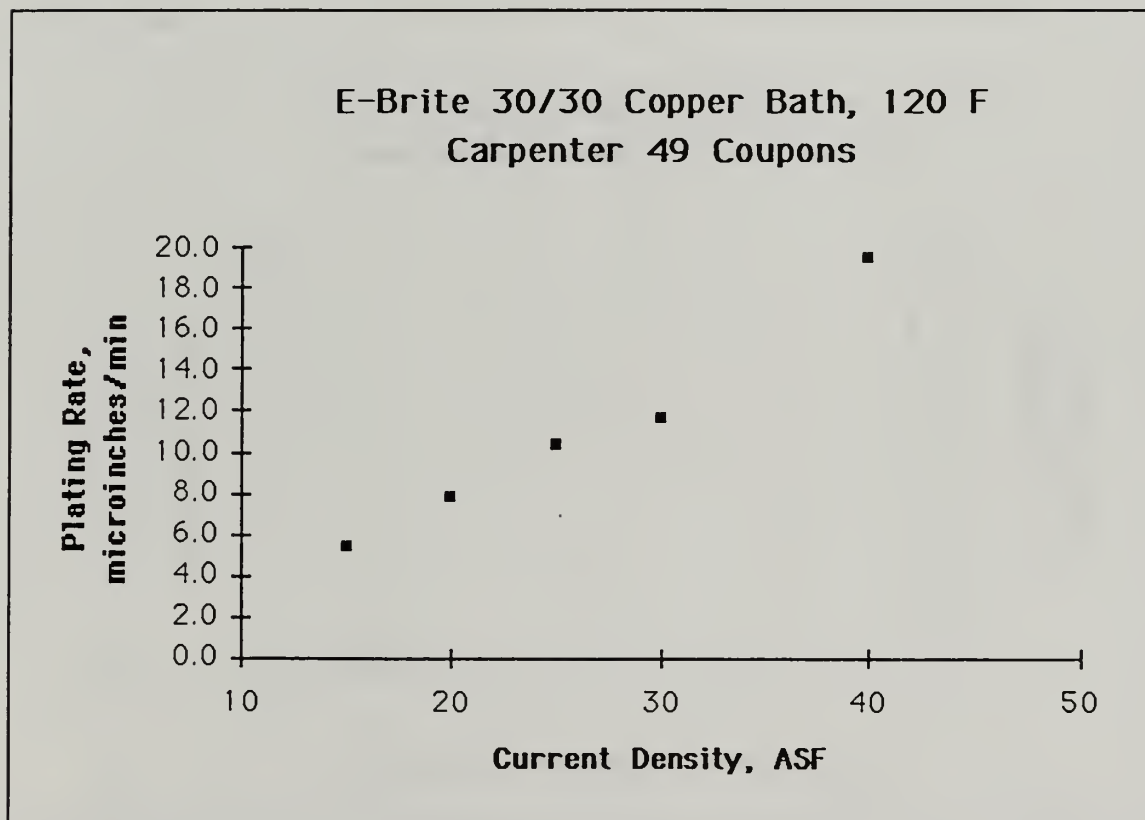


Figure 6 Kovar - Preliminary Coupon Testing in E-Brite 30/30 Bath

E-Brite 30/30 Bath

Bath Temp = 120 F \pm 10 F

Bath Time = 3 min

Sample #	Current Density ASF	Current m/a	Adhesion	Visual Appearance	Thickness uin	Estimated Plating Rate uin/min
1	15	120	pass	ok	20.8	6.9
2	20	160	pass	ok	28.0	9.3
3	25	200	pass	ok	38.4	12.8
4	30	240	pass	ok	50.9	17.0
5	40	320	pass	ok	60.0	20.0

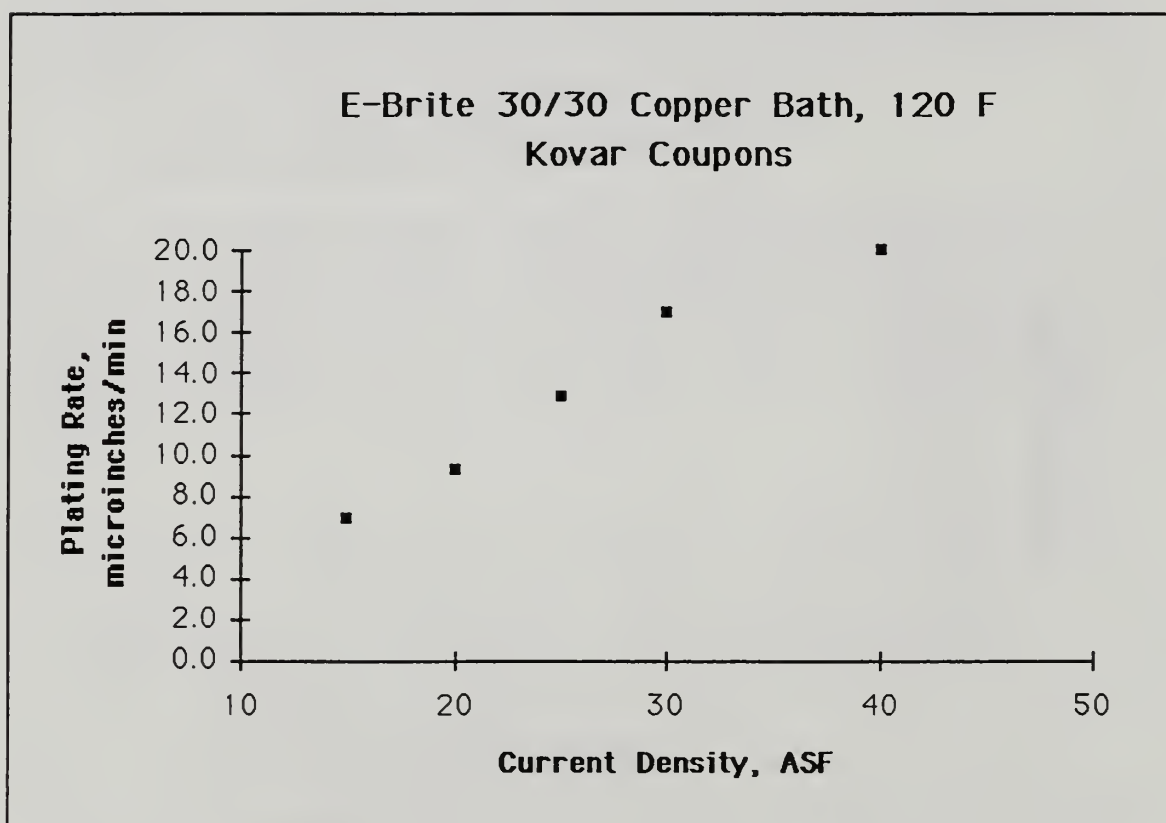


Figure 7 Aluminum - Preliminary Coupon Testing in E-Brite 30/30 Bath

E-Brite 30/30 Bath

Strike at 3 volts for 5 seconds

Bath Temp = 120 F \pm 10 F

Bath Time = 3 min

Sample #	Current Density ASF	Current m/a	Adhesion	Visual Appearance	Thickness uin	Estimated Plating Rate uin/min
1	15	405	pass	ok	30.3	10.1
2	20	540	pass	ok	37.6	12.5
3	25	675	pass	ok	52.4	17.5
4	30	810	pass	ok	60.3	20.1
5	40	1080	pass	burning	72.9	24.3

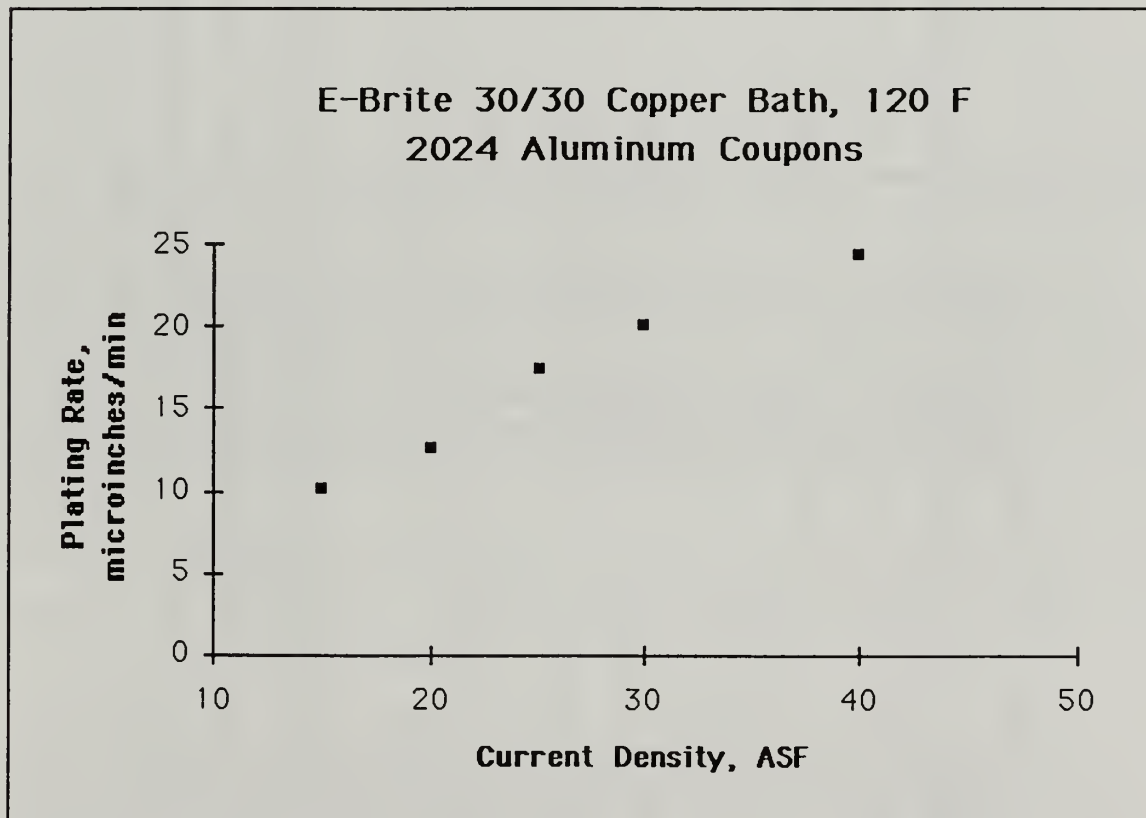


TABLE 2: RESULTS OF COPPER PLATING BATH COUPON STUDIES

PLATING PARAMETERS										PLATING RESULTS			
Material	No. of Samples	Sample Area, sq ft	Bath Type	Bath Temp, F	Current/ sample m/a	Current Density ASF	Time Min.	Adhesion	Visual Appearance	Average Thickness uln	Ave. Plating Rate uln/min	Solderability w/o aging	Comments
Carpenter 49	5	0.004	E-Brite 30/30 500 ml Beaker	120	100	25	9	pass	pass	110	12.2	pass	plated individually
Carpenter 49	5	0.004	CuCN 11 Liter Bath	120	200 100	50 25	5 5	pass	pass	84	8.4	pass	All samples plated at once
Kovar	5	0.008	E-Brite 30/30 500 ml Beaker	120	200	25	7	pass	pass	112	16.0	pass	plated individually
Kovar	5	0.008	CuCN 11 Liter Bath	120	400 200	50 25	5 5	pass	pass	151	15.1	pass	All samples plated at once
Al 2024	6	0.014	E-Brite 30/30 11 Liter Bath	120	210 to 230	15 to 17	6	pass	pass	74	12.3	pass	Plated in Pairs
Al 2024	6	0.014	CuCN 11 Liter Bath	120	420 210	30 15	0.5 3	pass	pass	69	19.7	pass	Plated in Pairs

Test Methods

Adhesion - A grid was scribed onto the coupon using an exacto blade. Tape was then applied to the grid area. Subsequently, it was rapidly removed. The plating was examined for lifting. This is similar to the method described in ASTM B571-79 paragraph 13.

Visual Appearance - The coupon was examined under a microscope at approximately 10X. Any evidence of blisters, peeling, or pitting were noted

Thickness - Thicknesses were measured using X-Ray Fluorescence on a Twintest XRF-PTG utilizing calibration standards.

Solderability - Solderability was evaluated per the guidelines of ASTM B678 with the exception that the coupons remained in the solder pot for longer periods of time.

long by 0.3 inches in diameter.

Both the copper cyanide and E-Brite 30/30 baths were maintained at $120^{\circ}\text{F} \pm 5^{\circ}\text{F}$. The Kovar and Carpenter 49 coupons plated individually in the 500 ml beaker of the E-Brite 30/30 bath. All five coupons (Kovar and Carpenter 49) were plated at once in the 11 liter cyanide bath. The aluminum coupons were plated in pairs in both the E-Brite 30/30 and copper cyanide 11 liter baths. Table 2 lists both the plating conditions and the results for these coupons. The chosen plating conditions were sufficient to yield coating thicknesses within the ranges specified for the individual parts. Table 3 contains the thickness specifications.

Table 3 Thickness Specifications

<u>Part Material</u>	<u>Coupon Material</u>	<u>Copper Thickness Spec., uin</u>
6061 Aluminum	2024 Aluminum	30 to 100
Alloy 52	Carpenter 49	60 to 200
Kovar	Kovar	30 to 200

Adhesion, visual appearance, solderability, and thicknesses were measured on all of the coupons.

Adhesion was evaluated by scribing a grid on the coupons with an exacto blade. A piece of tape was then applied to the area and rapidly lifted off at a 90° angle. Any lifting or peeling of the coating was cause for rejection. This method is similar to the method described in ASTM B571-79, Paragraph 13, "Standard Test Methods for Adhesion of Metallic Coatings". All of the coupons passed adhesion testing.

Visual appearance was examined under a microscope at 6 to 10 X. Any evidence of blisters, peeling, or pitting were causes for rejection. None of the coupons displayed these undesirable conditions.

Solderability was appraised by following the guidelines of ASTM B678-80, "Standard Test Method for Solderability of Metallic-Coated Products", with the following exceptions; The samples were not aged and the coupons were held in the solder pot for longer than the specified 5 seconds due to their relatively large masses. The samples were considered acceptable if the solder coating was adherent, bright, and uniform over at least 95% of the tested surface. Both the E-Brite 30/30 and the copper cyanide pieces which were

tested fulfilled the requirements.

Coating thicknesses were measured using X-Ray Fluorescence on a Twintest XRF-PTG Model 6000 utilizing appropriate calibration standards. This method is described in ASTM A754-79, "Standard Test Method for Coating Thickness by X-Ray Fluorescence". The average thickness are reported in Table 2. Table 4 provides the average individual coupon thicknesses. Approximately 10 thickness measurements were executed on each coupon. From the measurements, the E-Brite 30/30 seems to be experiencing a greater range in thicknesses than the copper cyanide bath. This appears to be a trait of the E-Brite 30/30 bath.

C. Aluminum Part Testing

Based on our favorable results with coupons, we proceeded to evaluate actual 6061 aluminum ribbon. These tests were performed in the 11 liter E-Brite 30/30 bath. Prior to plating the actual parts, the 11 liter bath was analyzed for pH, copper content, and electrolyte content to verify the analysis methods and ensure the proper chemistry of the bath.

The pH was measured with a Cole Parmer Model 598580 electronic pH meter with automatic temperature compensation. The meter was calibrated with NIST traceable standards prior to use. The starting pH of the bath was 8.5, a little lower than EPI's recommended range of 9 to 10.5. The technical literature notes that the pH will eventually increase with use into the recommended operating range.

A Perkin Elmer Model 6500 Inductively Coupled Plasma Atomic Emissions Spectrometer (ICP-AES) measured the copper content of the bath at 1.05 oz/gal (7.93 g/l). The analysis method is described in Appendix B. A titration method documented in the EPI technical literature or Atomic Absorption Spectroscopy may also be used to evaluate the copper level. This copper concentration measured was within EPI's range of 1.0 to 1.5 oz/gal.

A titration was executed to measure the E-Brite 30/31 electrolyte concentration. Appendix B contains a description of this technique. 42.2% by volume electrolyte was measured. According to the data in the EPI technical literature, presented graphically in Figure 8, the corresponding minimum % electrolyte expected for a copper concentration of 1.05 oz./gal (7.93 g/l) is 42.4%. We did not correct for the 0.2% deficit prior to plating our parts.

At this time we did not test for metallic contaminants since

Table 4: Comparison of Copper Coating Thicknesses

Coupon Material	Bath Type	Sample No.	Thickness Mean	Std Dev	Minimum	Maximum	Range
Carpenter 49	E-Brite 30/30 500 ml Beaker	1	107	14	90	123	33
		2	107	4	103	114	11
		3	99	8	84	108	24
		4	114	10	100	134	34
		5	124	8	110	141	31
		Overall	110	12	84	141	57
Carpenter 49	Copper Cyanide 11 Liter Bath	1	89	10	75	105	30
		2	76	5	69	85	16
		3	80	6	72	91	19
		4	91	5	83	102	19
		5	83	11	65	101	36
		Overall	84	10	65	105	40
Kovar	E-Brite 30/30 500 ml Beaker	1	97	5	89	103	14
		2	116	10	98	138	40
		3	105	8	89	117	28
		4	122	16	107	153	46
		5	118	10	106	136	30
		Overall	112	14	89	153	64
Kovar	Copper Cyanide 11 Liter Bath	1	155	7	143	168	25
		2	156	15	143	192	49
		3	165	15	140	191	51
		4	153	6	139	161	22
		5	128	8	111	136	25
		Overall	151	10	111	192	81
2024 Aluminum	E-Brite 30/30 11 Liter Bath	1	65	5	56	72	16
		2	61	7	50	70	20
		3	90	16	76	131	55
		4	84	8	72	103	31
		5	63	18	41	95	54
		6	83	9	76	103	27
		Overall	74	16	41	131	90
2024 Aluminum	Copper Cyanide 11 Liter Bath	1	66	5	60	75	15
		2	70	7	61	86	25
		3	71	8	64	89	25
		4	71	8	64	89	25
		5	66	9	58	88	30
		6	70	6	65	85	20
		Overall	69	7	58	89	31

our bath was essentially new. In the future we will probably check for iron, nickel, and zinc by ICP. This analysis will be performed concurrently with the copper content ICP analysis. According to EPI, metallic and organic impurities would require additional electrolyte (E-Brite 30/31) over and above that required for the copper concentration. This required additional electrolyte addition can be determined utilizing a Hull cell.

Our first plating attempt in the E-Brite 30/30 bath resulted in a fair looking ribbon with good adhesion, and the correct thickness but some darkened areas. The bath was maintained at 120°F and the parts were plated at a current density of approximately 15 amperes per square foot (ASF). These darkened areas were evident on both the ribbon itself and its 1100 alloy aluminum fixture.

Figure 1 displays the ribbon coiled on its fixture. The fixture is a .032 inch diameter 1100 aluminum rod. Figure 9 presents the darkened areas on the fixture at optical magnification levels. Figure 10 provides a Scanning Electron Microscope (SEM) photo. At this higher magnification, the darkened areas appear rougher and contain a copper oxide while the bulk plating of the rod pictured in Figure 11 appears uniform.

As a result of the findings, we decided to plate dummy fixtures to isolate the cause of the darkened areas. At first, we suspected that entrapment of plating or cleaning solutions at the surface induced by inadequate rinsing was the culprit. Subsequently, we improved our rinsing and drying techniques which partially enhanced the appearance of the rod but did not alleviate the problem. Next, we surmised that inadequate exchange of the solution was occurring at the surface of the parts. As a result we bubbled bottled welding grade nitrogen through the bath. This eliminated the darkened areas.

After the success of our fixture plating experiment, we plated one heat treat batch of the ribbon. This corresponds to 3 groups of five rods each or approximately 150 to 180 finished assemblies. One group is plated at a time, so we essentially performed three runs. We maintained all of our current surface cleaning and preparation operations only altering the actual plating bath in which the parts are plated. Table 5 presents the plating conditions and the results for each group of ribbon.

When examined at approximately 10X, none of the parts displayed any evidence of blisters, peeling, or pitting. No signs of damage to the aluminum ribbon were observed. The adhesion was evaluated on one sample from each group. A

Figure 8 E-Brite 30/31 Min. % Required for Copper Level

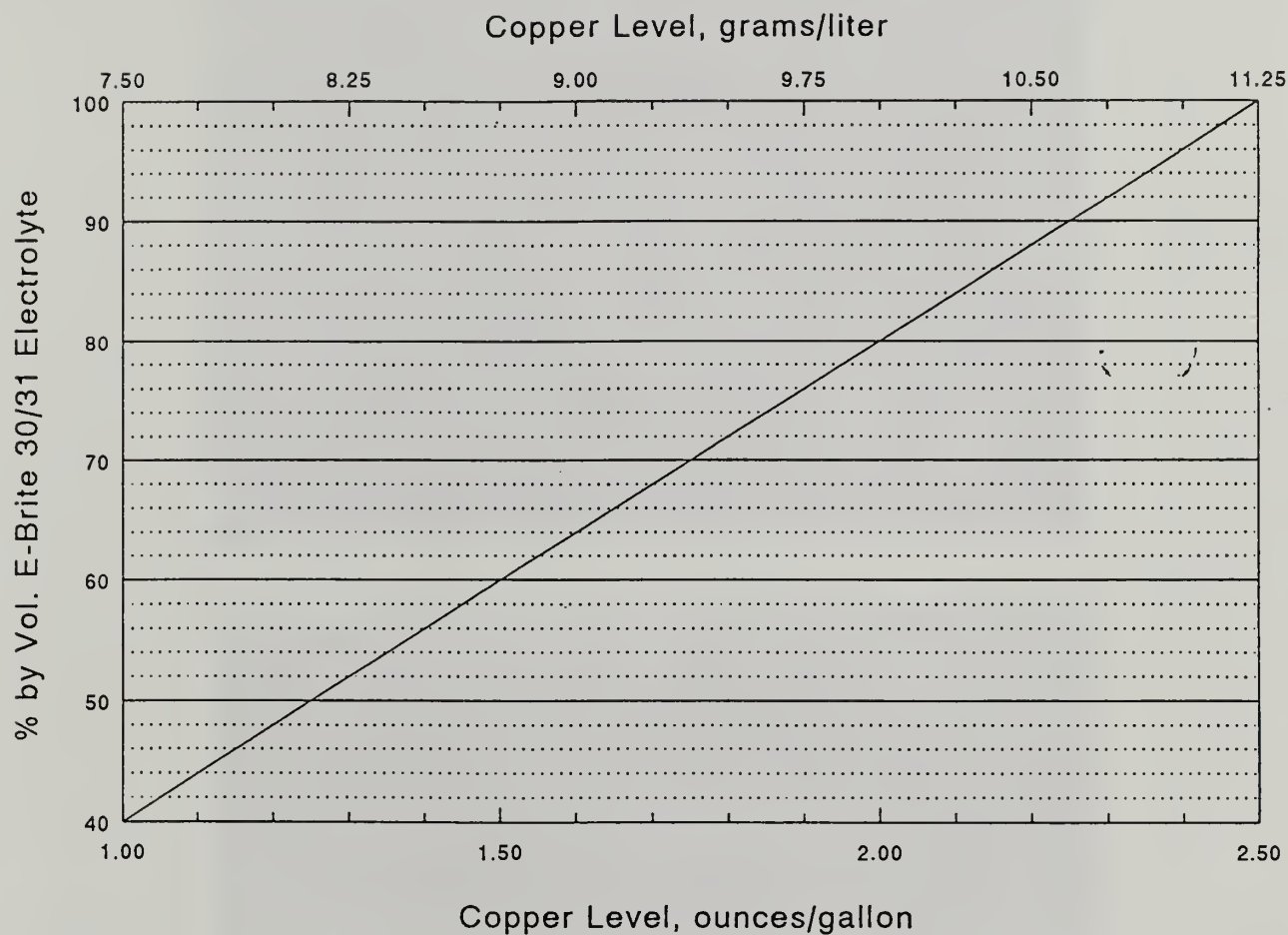
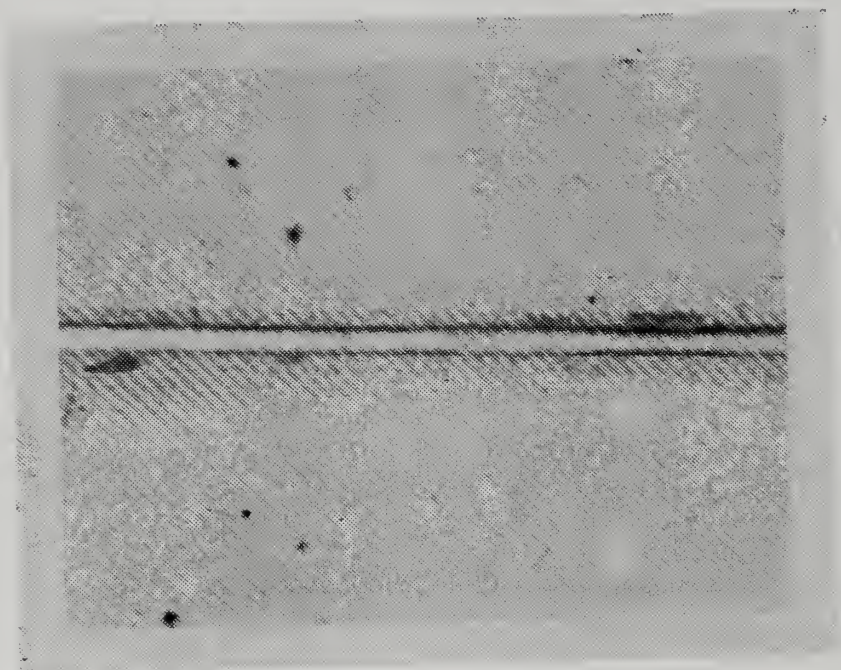
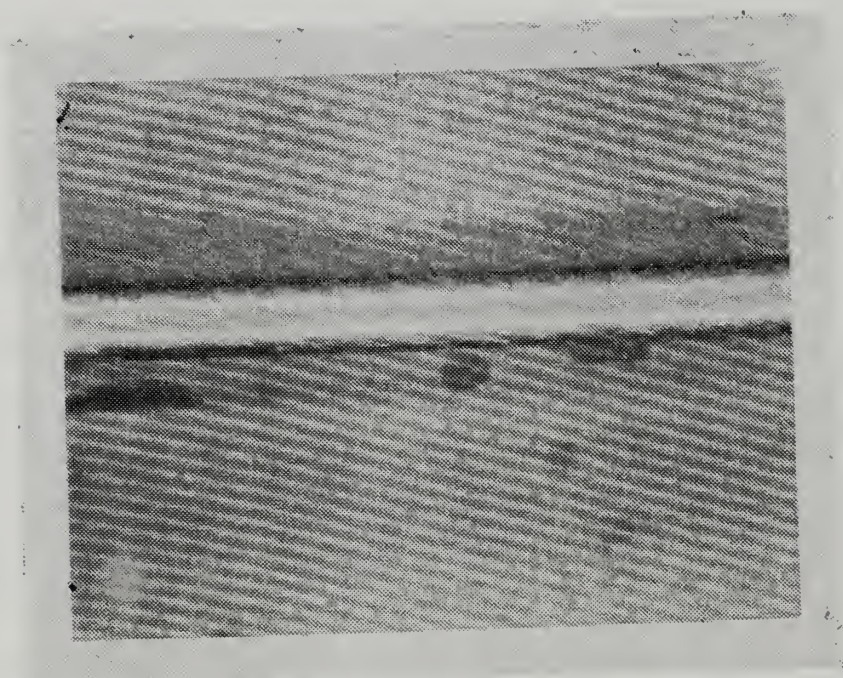


Figure 9 Darkened Areas on E-Brite 30/30 Cooper Plating on
Aluminum 1100 Rod



a. 9.6x

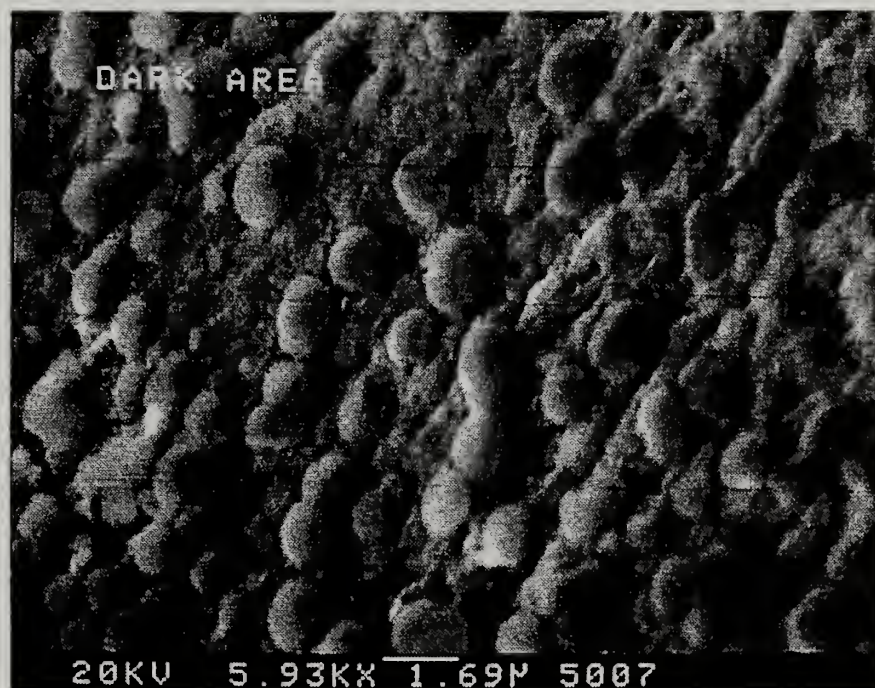


b. 25.6X

Figure 10 Darkened Areas on E-Brite 30/30 Cooper Plating on
Aluminum 1100 Rod



a. 448X



b. 5930X

Figure 11 Bulk E-Brite 30/30 Copper Plating on Aluminum 1100 Rod

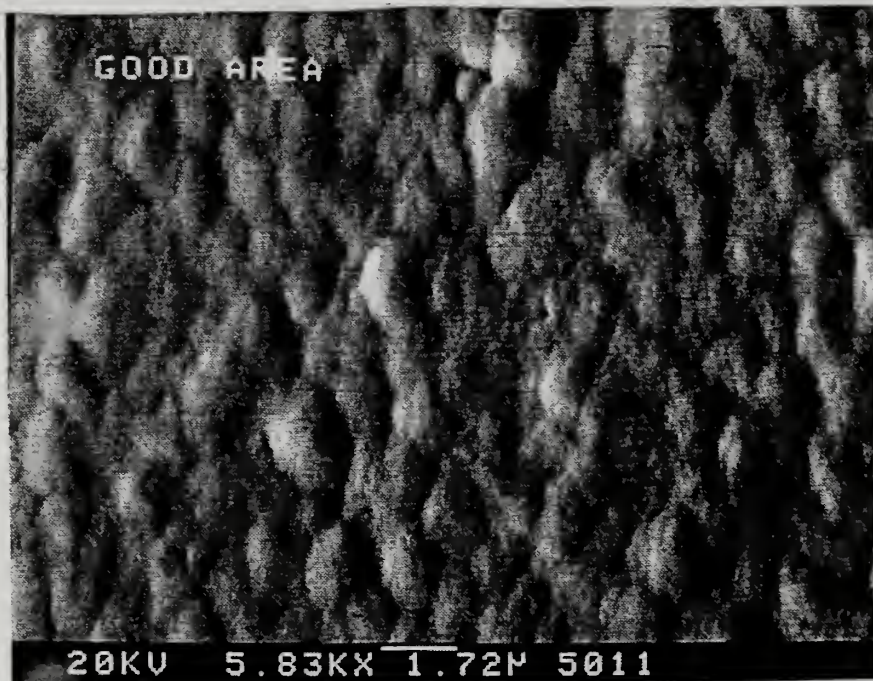


Figure 12 E-Brite 30/30 Copper Plated Aluminum Ribbon

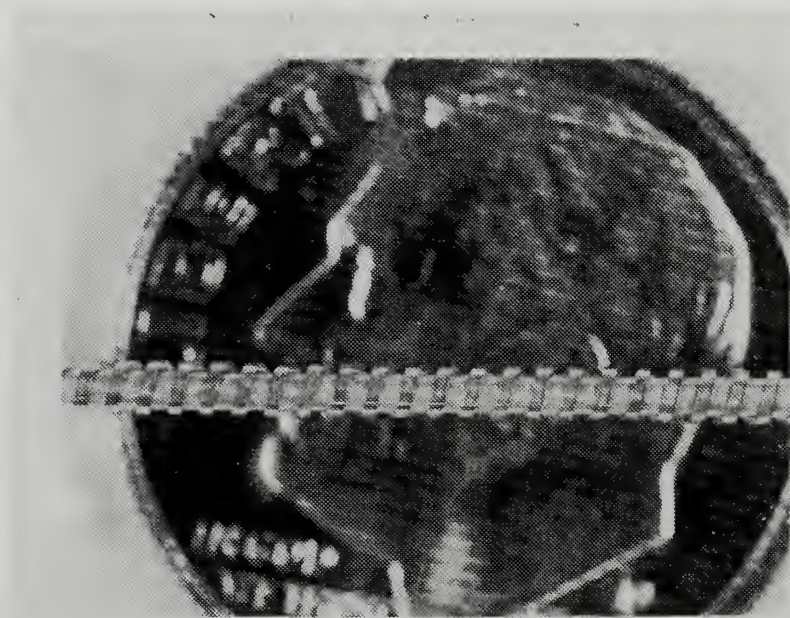


TABLE 5: RESULTS OF 6061 ALUMINUM RIBBON PLATING IN E-BRITE 30/30 COPPER BATH

PLATING PARAMETERS					PLATING RESULTS		
Group	Sample Area, sq ft	Current/ Group m/a	Current Density ASF	Time Min.	Adhesion	Visual Appearance	Average Thickness uin
A 5 rods	0.027 (.0051 per flexlead)	320	12	5.8	pass	pass	67
B 5 rods	0.027 (.0051 per flexlead)	320	12	6.3	pass	pass	79
C 5 rods	0.027 (.0051 per flexlead)	320	12	5.8	pass	pass	64

Test Methods

Adhesion – A piece of masking tape was applied to a sample of aluminum ribbon from each group. Using tweezers, the ribbon was rapidly removed. The plating was examined for lifting.

Visual Appearance – Every rod of ribbon was examined under a microscope at approximately 10X. Any evidence of peeling, blisters, or pitting were noted.

Thickness – Thicknesses were measured on all rods using X-Ray Fluorescence on a Twintest XRF-PTG utilizing calibration standards.

TABLE 6: COPPER PLATING THICKNESSES ON 6061 ALUMINUM RIBBON – E-BRITE 30/30 BATH

E-Brite 30/30 Bath

Temperature = 120 F

Current per Group = 320 milliamperes

Current Density = 12 ASF

PLATING THICKNESS RESULTS					
Group ID	Thickness, uin			Overall for Group	
Plating Time	Sample No.	Reading 1	Reading 2		
A 5.8 min.	1	82	66	Average Thickness	67
	2	65	66	Standard Deviation	12.8
	3	54	83	Minimum	49
	4	56	85	Maximum	85
	5	66	49	Range	37
B 6.3 min.	1	63	93	Average Thickness	79
	2	96	56	Standard Deviation	16.4
	3	94	78	Minimum	53
	4	81	87	Maximum	96
	5	93	53	Range	43
C 5.8 min.	1	68	78	Average Thickness	64
	2	78	73	Standard Deviation	12.6
	3	58	42	Minimum	42
	4	52	59	Maximum	78
	5	78	58	Range	36

Thickness - Thicknesses were measured using X-Ray Fluorescence on a Twintest XRF-PTG utilizing calibration standards.

**TABLE 7: COPPER PLATING THICKNESSES ON 6061
ALUMINUM RIBBON – COPPER CYANIDE BATH**

PLATING THICKNESS RESULTS					
Lot ID	Sample No.	Thickness, μin		Overall for Group	
		Reading 1	Reading 2		
9L7U	1	86	77	Average Thickness	70
	2	61	80	Standard Deviation	9.5
	3	74	66	Minimum	53
	4	66	69	Maximum	86
	5	65	53	Range	32
9L7V	1	62	62	Average Thickness	63
	2	69	59	Standard Deviation	4.7
	3	60	60	Minimum	55
	4	66	68	Maximum	69
	5	67	55	Range	14
9L7X	1	72	69	Average Thickness	68
	2	74	66	Standard Deviation	11.3
	3	68	75	Minimum	40
	4	83	74	Maximum	83
	5	40	64	Range	43

Thickness - Thicknesses were measured using X-Ray Fluorescence on a Twintest XRF-PTG utilizing calibration standards.

piece of ribbon was placed on some masking tape. It was then removed rapidly. No lifting of the plating was observed.

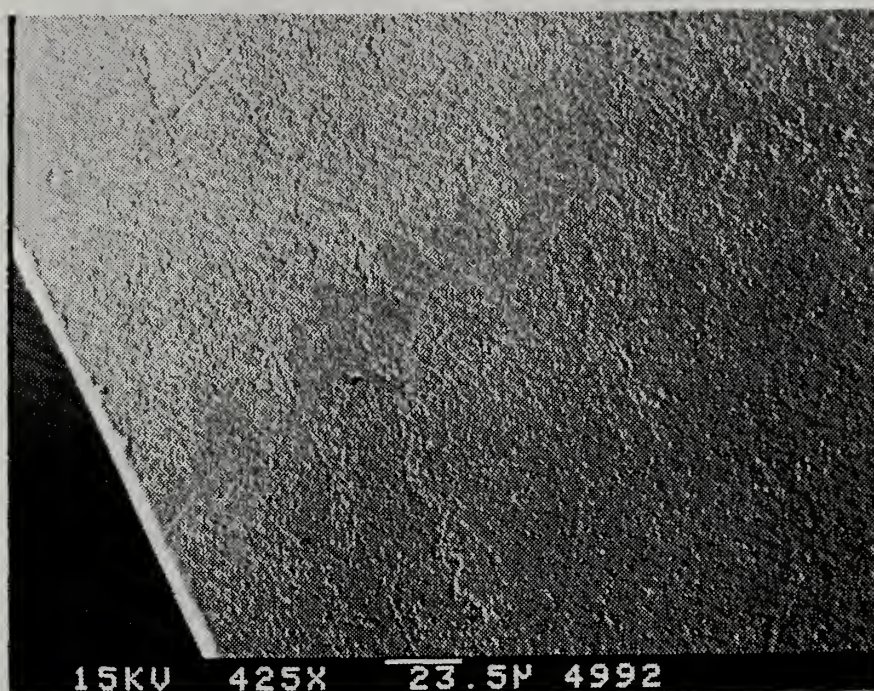
The thicknesses were measured by X-Ray Fluorescence. Two measurements were acquired per aluminum ribbon. Table 6 contains the thickness measurements. Some recent production data from the copper cyanide bath is included in Table 7 for comparison purposes. The aluminum ribbon like the coupons experienced a greater variability in thicknesses in the E-Brite 30/30 bath than the copper cyanide bath.

A sample of ribbon from one rod of each group was examined by SEM. Figure 12 displays a low magnification view, 5.4X, of the E-Brite 30/30 ribbon. Figures 13 through 15 present SEM photographs of a representative area of surface from each of the samples. Figure 16 provides a comparison with a copper cyanide plated ribbon. Both the E-Brite 30/30 and the copper cyanide coatings displayed some pin hole defects at magnifications of approximately 5000 to 6000 X. The E-Brite 30/30 appeared to display a slightly larger number of these defects in the areas examined.

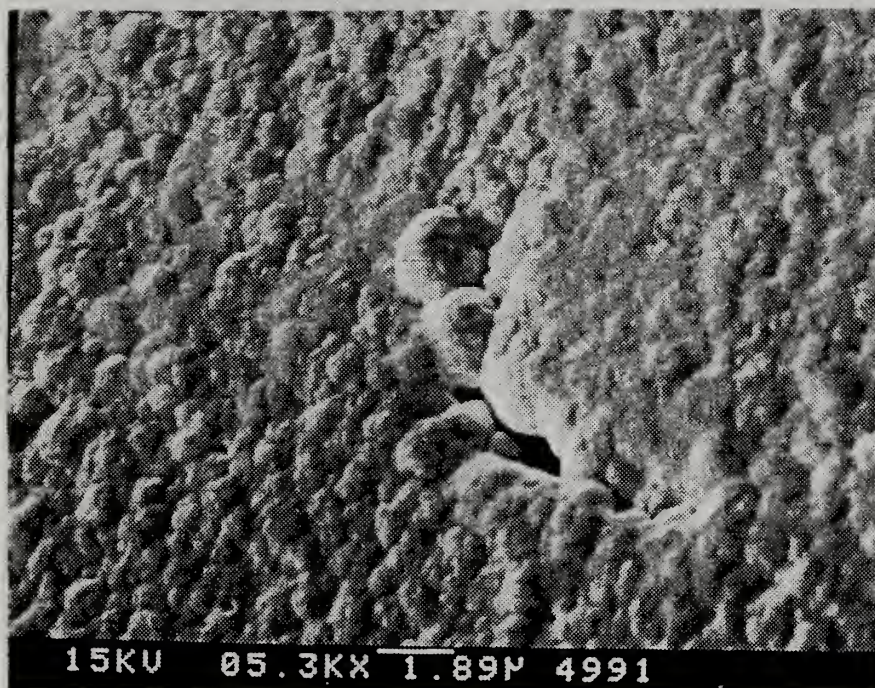
One rod from each group was fabricated into flexlead assemblies. See Figure 3 for the assembly configuration. These rods experienced the full manufacturing process. First, they were cut to size and soldered to beryllium copper clips utilizing an RMA Flux, ALPHA 611. No difficulties were experienced in this step. A total of 36 assemblies, 12 from each rod were produced. Next, the exposed copper coating was electrolytically stripped in a chromic acid solution. Subsequently, the excess ribbon on the assembly experienced a pull test to ensure the bond strength of the solder joint. All of the samples survived this test. Finally, the assemblies were inspected at a magnification of 8X for copper removal, ribbon surface condition, and appearance of solder joints. Thirteen of the assemblies were rejected for insufficient copper removal. These thirteen assemblies then experienced a second stripping operation to remove all of the copper. This corresponds to approximately 26 to 40 seconds total combined time for both the first and second stripping operation. Our current process allows for double stripping i.e., 26 seconds total within the stripping solution. All of these assemblies passed a second inspection.

Next, the completed assemblies were examined by SEM. Particular attention was paid to the aluminum ribbon. Figures 17 through 19 display the aluminum ribbon from an E-Brite 30/30 stripped assembly, an E-Brite 30/30 double stripped assembly, and a copper cyanide stripped assembly. All of these ribbons exhibit grain boundary etching.

Figure 13 E-Brite 30/30 Copper Coating on 6061 Aluminum Ribbon -
Group A



a. 425X



b. 5300X

Figure 14 E-Brite 30/30 Copper Coating on 6061 Aluminum Ribbon -
Group B



a. 443X

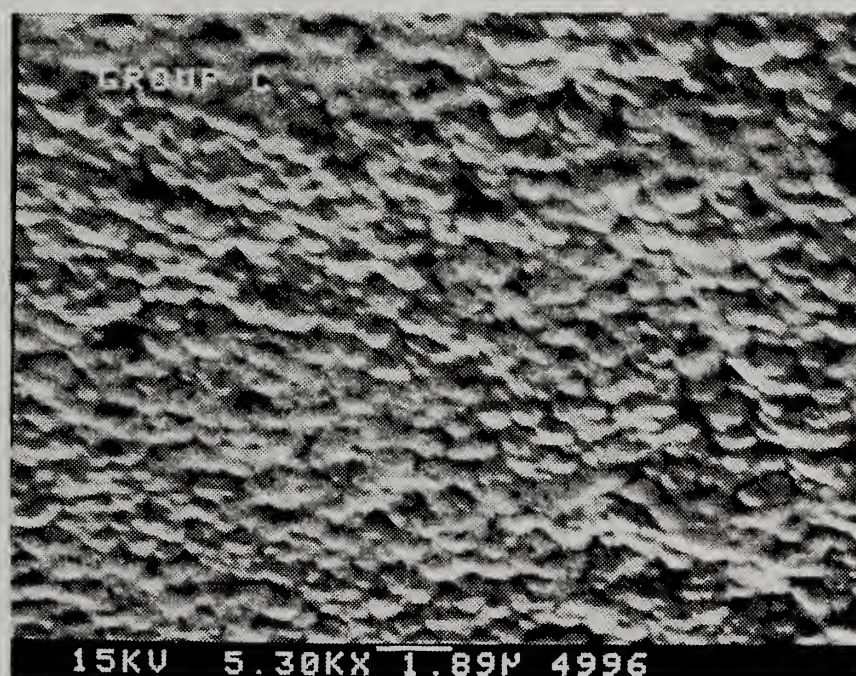


b. 5300X

Figure 15 E-Brite 30/30 Copper Coating on 6061 Aluminum Ribbon -
Group C



a. 443X

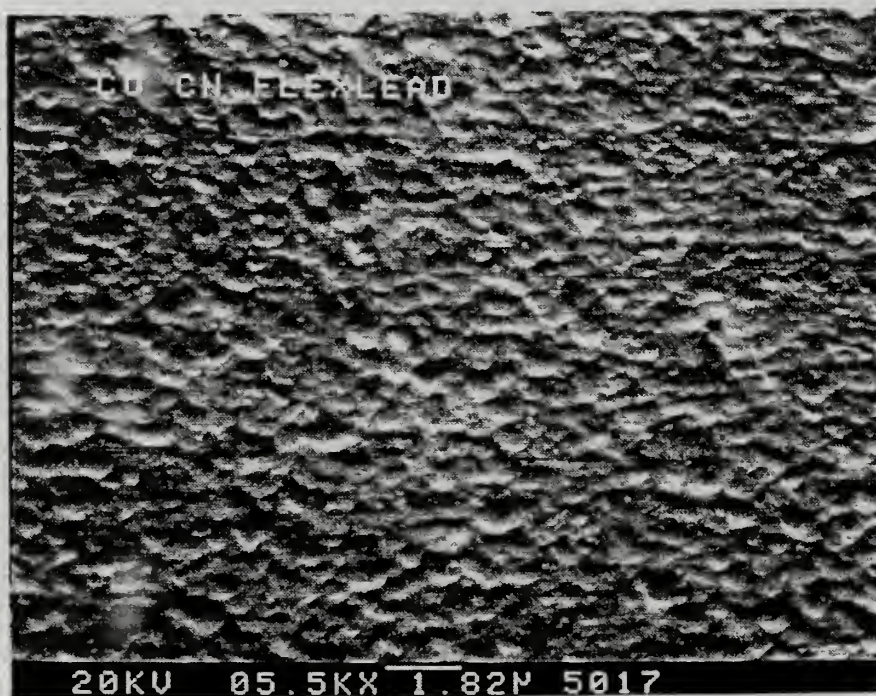


b. 5300X

Figure 16 Copper Cyanide Coating on 6061 Aluminum Ribbon



a. 429X



b. 5500X

Figure 17 6061 Aluminum Ribbon in E-Brite 30/30 Assembly - After
1 Stripping Cycle of 13 Seconds



a. 475X



b. 5330X

Figure 18 6061 Aluminum Ribbon in E-Brite 30/30 Assembly - After
2 Stripping Cycles from 26 to 40 Seconds Total



a. 443X

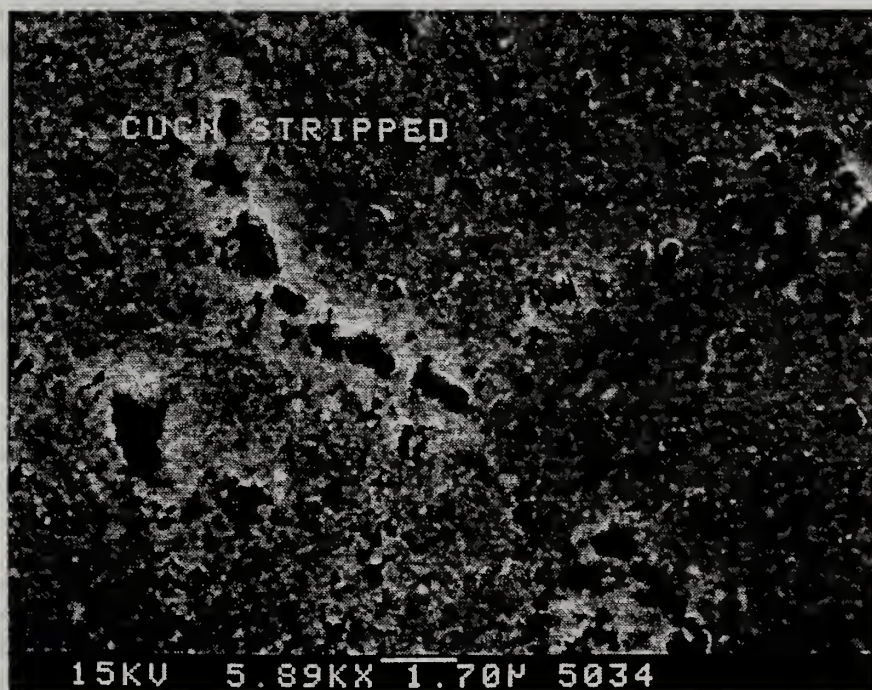


b. 5900X

Figure 19 6061 Aluminum Ribbon in Copper Cyanide Assembly -
After a Stripping Cycle of 13 to 26 Seconds



a. 443X



b. 5890X

The completed assemblies were then submitted to two additional qualification tests, electrical testing and solvent cleaning testing.

The electrical testing involved steadily increasing the current through the flexlead assemblies until failure. Three flexleads from each group were immersed in a bromotrifluorethylene damping fluid. A 800 hertz current starting at 1 ampere was applied for 10 minutes. The current was increased in increments of .25 amperes and repeated until 3 amperes was reached. No fusing of any of the flexlead assemblies was observed. The current was then slowly increased until fusing was observed.

The flexleads from all of the groups experienced fusing at 3.5 amperes or above. The E-Brite 30/30 flexlead assemblies, regardless of stripping time, demonstrated equivalent current carrying properties to the copper cyanide assemblies. Therefore, the E-Brite plating process produced no negative electrical property effects.

The solvent cleaning test was performed in a Crest 1410 ultrasonic degreaser at a power of 75 ± 5 watts for one minute in freon 113. This is the most aggressive cleaning that the flexlead assemblies would encounter in our production line. All of the assemblies endured the cleaning process with no visual damage at 10X.

All of the comprehensive testing of the E-Brite 30/30 copper plating process on the zincated aluminum flexleads has produced positive results. The E-Brite 30/30 copper plating process has met our primary requirements of good adhesion, acceptable appearances, and solderability. Some difficulties in stripping the deposit were encountered. However, these stripping problems are not unusual. They occur periodically with the current copper cyanide process. Our primary concern with stripping the assembly for longer periods of time involves not the flexlead but the beryllium copper clips to which they are soldered. These clips are attacked during the stripping process. Too extensive a duration in the stripping solution can alter the dimensions of the clips rendering the entire assembly worthless.

D. Kovar Part Testing

Our major technical requirements for the Kovar feedthrus are for good adhesion of the coating and compatibility of the plating solutions with the glass and glass to metal seals. Photographs of these parts are presented in Figure 20. These parts are used to seal an area in our gyro. Electrical leads are conveyed through the holes in the pins on this feedthru header. The leads are then soldered into

place and an airtight seal is formed. The pins are Kovar, a nickel-iron-cobalt glass sealing alloy. The interior surface of the pin is solder coated to promote good sealing properties. To achieve this solder coating, we barrel electroplate the parts in copper cyanide to provide good adhesion for a subsequent solder electroplating process.

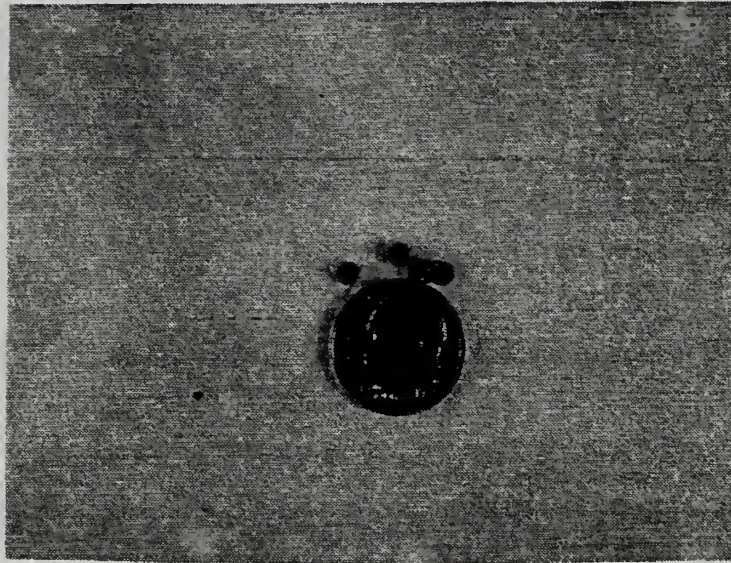
The original copper plated Kovar coupons that we produced demonstrated good adhesion. We have no reason not to expect the actual parts to exhibit the same qualities. As mentioned above the copper plating step is followed by immediate plating in a 60/40 tin lead solder bath. This bath is fluoborate based and has a high tolerance for copper, iron and nickel contamination. No problems with the E-Brite 30/30 coating are anticipated with this solder bath.

The Kovar parts are currently plated in a barrel for 65 minutes in the copper cyanide. Both time constraints and the desire to keep our E-Brite bath cyanide free limited our ability to attempt to develop the actual barrel plating process. Cyanide poisons the E-Brite 30/30 bath. We only possess one plating barrel which is used for production. Destroying the cyanide is quite a lengthy process. It would require completely washing the barrel with sodium hypochlorite then soaking it in a tank of 2% sodium hypochlorite for a minimum of 24 hours. Next, water rinsing followed by a 2% sulfuric acid rinse, followed by a water rinse succeeded by another water rinse, a 5% potassium hydroxide rinse and more water rinsing would be necessary. In light of these considerations, we decided to hand plate a small quantity of these parts in the E-Brite 30/30 bath.

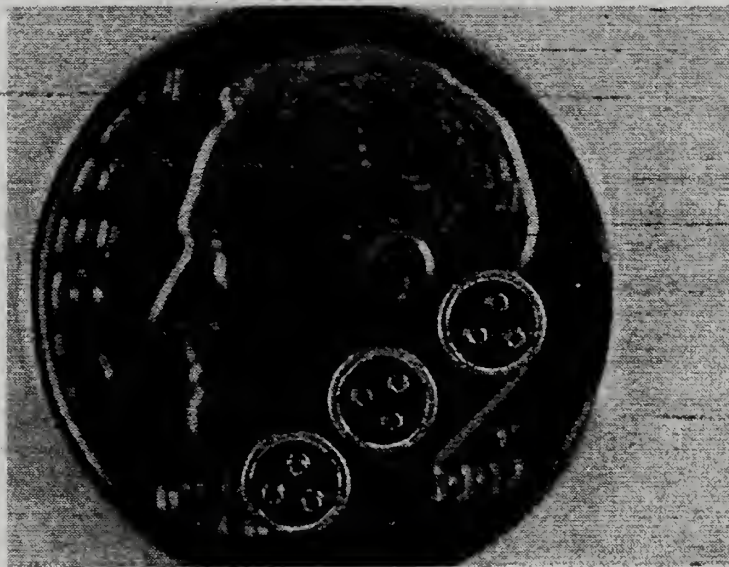
Twenty feedthru headers and some plating balls were plated in the E-Brite 30/30 bath at $120^{\circ}\text{F} \pm 5^{\circ}\text{F}$ and 1.5 amperes for thirty minutes. Hand agitation of the parts and balls in their metal basket was provided. Bottled nitrogen was bubbled through the bath. This was not a simulation of the barrel process but an attempt to determine the corrosivity of the solution with respect to the glass and the glass to metal seals.

After plating, the thicknesses of the deposits on the pins and outer diameter were measured by X-Ray Fluorescence. We require 30 to 200 microinches on the pins. Only 8 microinches were deposited on the pins while approximately 80 microinches were deposited on the outer diameter. The visual appearance and adhesion of the deposit were excellent. Next to ensure the integrity of the glass and the glass to metal seals, we helium leak checked the device. The holes in the pins were sealed with an epoxy. The parts

Figure 20 Kovar Feedthru Header



a. Approx.
Actual Size



b. 4.3X

Figure 21 Feedthru Header with Alloy 52 Pins



were then mounted in a specially designed fixture onto a helium leak detector. No leaks in excess of 1×10^{-8} cc Helium/sec/atm at room temperature were detected. No damage to the glass or glass to metal seals had occurred during our plating process.

All of the results from our limited plating tests with Kovar and the E-Brite 30/30 copper bath are favorable. The deposit displays good adhesion. The integrity of the glass and glass to metal seals remains undamaged after completion of this process.

E. Alloy 52 Part Testing

Currently, the plating of this feedthru header is being performed at our suppliers. This supplier plates the Alloy 52 pins on the header pictured in Figure 21 with nickel prior to solder plating them. In the event that this process needed to be performed on site, we are confident from our experience with plating the Carpenter 49 coupons and the Kovar glass compatibility tests that the E-Brite 30/30 Copper could successfully be applied while maintaining both good adhesion and the integrity of our glass to metal seals.

V. TOXICS USE REDUCTION ASSESSMENT

This project demonstrates that it is technically feasible to copper electroplate a variety of delicate parts without the need for cyanide-based electroplating solutions. The replacement of the cyanide-based electroplating bath with the E-Brite 30/30 bath would reduce health and environmental risks.

The composition of the E-Brite bath is essentially non-hazardous although standard precautions and protective equipment as are generally used in a metal finishing operation should be employed. However, the special precautions and equipment that are necessary working with cyanides are no longer needed. Thus antidote kits, decontamination solutions and pre-arrangements with the local hospital are no longer required.

The protection of workers from accidental exposure to highly toxic cyanides will be one of the greatest benefits to converting to the E-Brite process.

In addition to the elimination of cyanides from the workplace the following benefits will also be realized:

- By-product generation in the form of concentrated solutions, rinsewaters and air emission of cyanides are completely eliminated.
- Treatment of the E-Brite rinsewaters requires only pH adjustment and precipitation. It contains no chelating agents. This eliminates the need for the two-step cyanide destruction process with hypochlorite.
- The operational life of the E-Brite bath is estimated to be the same as a cyanide bath. The E-Brite bath can be maintained with electrolyte and copper additions.
- Existing wetted equipment can be reused if decontaminated to remove all traces of cyanide. (Due to the small size of our operations, we intend to replace our existing equipment).

From a toxics use reduction standpoint, we found no reason not to convert our existing cyanide-based process.

VI. ECONOMIC ASSESSMENT

To assess the economic feasibility of the conversion process, we evaluated the differences between the two methods for aluminum plating and determined either the expense costs or labor hours involved. We found that we could realize saving indirect expenses, although additional labor hours were required to utilize the new process. It should be noted that as a result of the small size of our operations, the absolute cost differences are not as important as they might be in a large scale plating operation.

A summary of the additional costs and savings is presented in Table 8. Further breakdown of the costs is presented in Table 9.

The significant factors affecting the economics of the conversion are:

- Hazardous waste disposal
- Labor reductions from reduced bath analysis primarily for carbonates and Rochelle salts.
- Increased labor for aluminum electroplating resulting from the slower plating speed of the E-Brite 30/30 process.

While the latter factor is the most significant in our process, it may be less so for parts requiring thicker deposits since additional work could be performed. In our aluminum operations we found the increase from one minute (cyanide bath) to six minutes (E-Brite) was too short to perform other activities. It remains our intention to convert to the non-cyanide based process for the workplace safety reasons discussed previously even though the costs of this conversion indicate no overall savings at our labor rates.

Table 8 Additional Costs/Savings of E-Brite 30/30 Process

<u>Direct Expenses</u>	<u>Annual Savings (Additional Costs)</u>
Plating Bath	\$ 50
Waste Disposal	570
Cyanide Antidote Kits	200
Filtration Supplies	(50)
Nitrogen (Note 1)	<u>(45)</u>
Net Savings	\$725/yr.

LABOR

Electroplating Aluminum (Note 2)	\$(3748)
Analytical	<u>2397</u>
Net Additional Cost	<u>\$(1351/yr)</u>
Total Additional Cost	\$(626/yr)

NOTES:

1. Nitrogen was used in lieu of clean dry air. Installation of a coalescing filter on a compressed air source would reduce costs.
2. Additional electroplating labor for the aluminum parts based on short (~6 minute) cycle which precludes concurrent activity.

Table 9 Economic Comparison of Copper Cyanide Bath
to E-Brite 30/30

Bath Make-Up - Chemicals for an 11 liter Bath

Copper Cyanide

E-Brite 30/30

462 gms CuCN @ \$97/kg
 550 gms NaCN @ \$55/kg
 330 gms Na₂CO₃ @ \$38/kg
 660 gms Rochelle Salts @ \$33/kg
 Deionized Water to Make Up

5.5 liters E-Brite 30/30 @ \$11/l
 Deionize Water to Make Up

Total Cost = \$110

Total Cost = \$61

Bath Analysis Frequency and Approximate Hours

pH, every lot, .25 hrs
 Free CN, every lot, .5 hrs
 Cu, monthly, 1 hr
 Carbonates, quarterly, 4 hrs
 Rochelle Salts, quarterly, 4 hrs

pH, daily when in use, .25 hrs
 Cu,Fe,Ni,Zn, monthly, 1 hr
 E-Brite 30/31, monthly, 1 hr

Disposal Cost of Dragout Rinse & Concentrate - 55 Gal. Drum

\$690

\$120

APPENDIX A Technical Data Sheets



E-Brite 30/30
30/31

TECHNICAL DATA

Electrochemical Products, inc.

E-Brite 30/30

17000 Lincoln Avenue New Berlin, WI 53151-2781 (414) 786-9330 FAX (414) 786-9403

Non-Cyanide Alkaline Copper Plating

E-Brite 30/30 plates directly on steel, brass, stainless steel, zincated aluminum and most high quality properly prepared zinc diecastings in both rack and barrel installations. Lead alloys can be plated in rack lines.

It eliminates the necessity of striking in cyanide copper.

E-Brite 30/30 eliminates potential health and environmental liability and the high cost of waste treating the cyanide.

Accidental drag-in of E-Brite 30/30 into an acid copper solution poses no health hazards due to evolution of poisonous cyanide gas as with cyanide copper.

The solution does not have to be treated for carbonates as with cyanide solutions.

E-Brite 30/30 produces a fine grained, smooth, dense and ductile copper deposit which is non-porous with excellent bonding properties. The plate may be buffed easily for a high luster.

It has excellent throwing and covering power.

It is used as a preplate for nickel and acid copper.

The E-Brite 30/30 plate is an excellent heat treat stop-off and EMI shield.

It is also an excellent decorative finish for buttons, rivets, etc. The copper plate can be readily blackened or oxidized for a variety of attractive antiqued finishes such as those found on wall plates, lighting fixtures and builders hardware.

Zinc diecast surfaces must be properly cleaned with EPI's E-Kleen 152 soak cleaner and activated with E-Pik 211 acid salt formulation. Some diecast surfaces may require anodic cleaning with E-Kleen 122.

E-Brite 30/30 process does not require an auxiliary tank and special expensive ceramic anodes for continuous dummy plating as other processes do. E-Brite 30/30 does not require coated anode baskets or the use of DI water as other processes do. The E-Brite 30/30 bath is stable and does not have to be periodically dumped and recharged as with other non-cyanide alkaline copper processes.

E-Brite 30/30 is supplied as a liquid concentrate which is diluted with tap water.

PLATING SPECIFICATIONS

RACK PLATING

	<u>Optimum</u>	<u>Range</u>
Concentration	50%	40 to 60%
E-Brite 30/30	by volume	by volume
Copper Metal	1.2 oz/gal.	1.0 to 1.5 oz/gal.
pH	9.5	9 to 10.5
Temperature	120°F	100 to 150°F
Cathode-Current Density	- -	15 to 30 ASF
Anode-Current Density	- -	5 to 15 ASF
Voltage	- -	1 - 6 Volts
Agitation	Vigorous Air	
Plate Thickness	Minimum of 0.0002 inches for strike	

BARREL PLATING

	<u>Optimum</u>	<u>Range</u>
Concentration	60%	50 to 75%
E-Brite 30/30	by volume	by volume
Copper Metal	1.5 oz/gal.	1.2 to 1.8 oz/gal.
pH	9.8	9.5 to 10.0
Temperature	140°F	120 to 150°F
Cathode-Current Density	- -	5 to 10 ASF
Anode-Current Density	- -	5 to 15 ASF
Voltage	- -	5 to 18 Volts
Agitation	2 - 6 RPM's and air agitation	

EQUIPMENT & OPERATION

Copper Anodes: OFHC or ETP bar stock

Anode/Cathode ratio: 1.5:1 **Note:** In barrel plating it is important to have the proper ratio. Calculate the maximum cathode area before setting up the process and insure that the anode area equals the maximum cathode area. Also it is required that the anode area be such that the anode current density is sufficient to corrode the anodes.

Filtration: Continuous 10 micron, 2-3 turns per hour. New filter cartridges must be flushed prior to use by recirculating warm water through the cartridges. A sulfur-free carbon pack must be maintained on the bath--one pound per 100 gallons - change weekly. When plating heavy copper (one (1) mil. or more) a 1 micron filter is recommended.

(over)

Heating: Stainless steel or titanium elements or coils.

Tank: Mild steel lined with rubber, koroseal or polypropylene or a drop in liner. All plastic tanks may be used. Large polypropylene tanks must be reinforced.

Ventilation: Not require, but it is a good practice as with all heated plating solutions.

Contamination: Lead and cyanide are contaminants - bath plates with black smut when contaminated.

PLATING ADDITIVES

E-Brite 30/30: Concentrate used in make-up of new baths and replenishment. Contains the copper metal and the other balanced components of the bath.

E-Brite 30/31: Electrolyte replenisher. Periodic adds over time -Adds determined by EPI.

E-Brite 30/32: High Current Density (HCD) area booster used with rack plating. Charge new rack baths with 3% by volume. Added thereafter as needed based on Hull cell tests for burn in HCD. For barrel installations EPI lab will recommend level of 30/32.

E-Brite 30/35: Buffer to control pH. Additions determined by pH measurement. Added to raise the pH upon bath make-up and when the pH decreases from acid drag in.

pH CONTROL FOR E-BRITE 30/30 PLATING SOLUTION

The pH will increase gradually as the bath is used and then stabilize in the range of 9 to 10.5. An electronic pH meter must be used. pH paper should be used only for spot checks. EPI will make recommendations for lowering pH, if necessary.

REPLENISHMENT OF PLATING SOLUTION

E-Brite 30/30 concentrate has a copper concentration of 2.4 oz/gallon. If the copper concentration in the bath decreases, additions of E-Brite 30/30 concentrate are required to replenish the copper and the other balanced components in the working solution. The strength of the working solution is monitored by determining the copper concentration. An anode to cathode ratio greater than 1.5:1 (example: 2:1) will cause the copper to climb in the bath, necessitating the frequent additions of E-Brite 30/31 electrolyte.

E-Brite 30/31 electrolyte is added on a regular basis (daily) to complex the copper dissolved from the anodes and to replace drag-out. It's consumption will depend upon drag-out, copper metal content and metallic contamination in the bath. The proper level of E-Brite 30/31 plating solution must be maintained for maximum adhesion to the base metal.

CLEANING PARTS

Unlike cyanide baths, the E-Brite 30/30 solution does not offer any cleaning. Therefore, it is extremely important to evaluate the cleaning in an existing line. Cleaners must be free-rinsing. EPI has soak and electrocleaners, as well as acid salts, that are compatible with the E-Brite 30/30 solution.

METALLIC COPPER ANALYSIS

1. Pipette a 5 ml. sample of the plating solution into a 250 ml. Erlenmeyer flask. Add 25 mls. distilled or deionized water.
2. Add 2 to 3 grams Ammonium Persulfate - let stand for 10 to 15 minutes. (Swirl a few times while waiting.)
3. Add approximately 5 mls. of Ammonia. Solution will be a clear deep blue.
4. Add 50 mls. distilled or deionized water.
5. Add 4 to 6 drops Pan Indicator. (Do not add more than 6 drops as it will affect the end point.)
6. Solution should be purple or pale red in color.
7. Titrate with 0.1M EDTA solution to a yellow-green end point.

Calculation: $\text{oz/gal. of copper} = (\text{mls. of EDTA}) \times 0.170$

Note: When E-Brite 30/30 copper is used as a replacement for a cyanide copper plating solution, the tanks, anodes and anode bars, baskets and barrels must be free of cyanide before charging the tank with the 30/30 solution. After the removal of the cyanide solution, the equipment should be washed with Hypochlorite solution and the tank soaked for 24 hours in 2% Sodium Hypochlorite to destroy residual cyanide. Do not neglect racks, barrels, heating and filtering equipment. After removal of the Hypochlorite solution and water rinsing, a dilute 1 to 2% Sulfuric Acid rinse should be used, followed with another rinse with cold water and then a 5% Potassium Hydroxide rinse should be used to eliminate the acid. Remove the alkaline rinse, rinse with cold water and then make up the tank with the E-Brite 30/30 solution. When destroying cyanide, forced ventilation should be used at all times, to prevent toxic cyanide fumes from accumulating. Personnel should be equipped with self contained breathing apparatus.

Best results are obtained with a new tank or by installing a new flexible liner in a tank which previously contained a cyanide copper plating solution. New anodes and baskets are also recommended.

WASTE TREATMENT

Copper from E-Brite 30/30 rinse water by itself or mixed with other metallic rinse waters is precipitated by conventional Sodium Hydroxide treatment with EPI Coagulants and EPI Polymers. A spill of E-Brite 30/30 requires treatment with lime.

CAUTION: The E-Brite 30/30 concentrate is a mild skin irritant. **DO NOT** work with E-Brite 30/30 without first reading and understanding the Material Safety Data Sheet furnished by EPI.

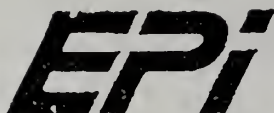
PACKAGING

Five (5) and 55 gallon non-returnable containers.

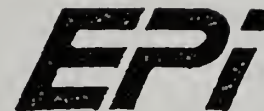
IMPORTANT NOTICE!

The following is made in lieu of all warranties, expressed or implied, including the implied warranties of merchantability and fitness for purpose: seller's and manufacturer's only obligation shall be to replace such quantity of the product as proved to be defective. Before using, user shall determine the suitability of the product for its intended use, and user assumes all risk and liability whatsoever in connection therewith. **Neither seller nor manufacturer shall be liable either in tort or in contract for any loss or damage, direct incidental or consequential arising out of the use or the inability to use the product.**

ATTN: Martha



MATERIAL SAFETY DATA SHEET



Section 1. Identity of Material

PRODUCT NAME OR NUMBER		E-Brite 30/30			
REGULATED IDENTIFICATION	DOT PROPER SHIPPING NAME	Compounds or Agents, Proprietary Electroplating Additives, Liquid			
	SHIPPING ID NUMBER	UN	- - -	NA	- - - DOT HAZARD CLASS NON-HAZARDOUS

Section 2. Hazard Specifications

Section 313 Supplier Notification: This product contains the following toxic chemicals subject to the reporting requirements of section 313 of the Emergency Planning and Community Right-To-Know Act of 1986 and of 40 CFR 372 or other hazards under 29 CFR 1910.1200.

CAS NUMBER	CHEMICAL NAME	% BY WEIGHT	PEL=	TLV=	CARCINOGEN
7440-50-8 as Cu	Copper, Basic Salt of: Section 313	< 4	1.0mg/M ³	1.0mg/M ³	NO
- - - -	No others listed in Section 313	- -	- - - -	- - - -	- - - -
- - - -	None listed in 1910.1200	- -	- - - -	- - - -	- - - -
- - - -	Water & other non-hazardous non-listed or non reportable materials	96	- - - -	- - - -	- - - -

KNOWN HAZARDS UNDER 29 CFR 1910.1200

	YES	NO		YES	NO
CORROSIVE MATERIAL		X	SKIN HAZARD		X
FLAMMABLE MATERIAL		X	EYE HAZARD		X
PYROPHORIC MATERIAL		X	TOXIC AGENT		X
EXPLOSIVE MATERIAL		X	HIGHLY TOXIC AGENT		X
UNSTABLE MATERIAL		X	SENSITIZER		X
WATER REACTIVE MATERIAL		X	REPRODUCTIVE TOXIN		X
OXIDIZER		X	BLOOD TOXIN		X
ORGANIC PEROXIDE		X	NERVOUS SYSTEM TOXIN		X
COMBUSTIBLE LIQUID		X	LUNG TOXIN		X
COMPRESSED GAS		X	LIVER TOXIN		X
IRRITANT		X	KIDNEY TOXIN		X
			RESPIRATORY SYSTEM		X

HMIS- HAZARD SIGNALS

HEALTH	1	FLAMMABILITY	0
REACTIVITY	0	PERSONAL PROTECTION	C
0=Minimal 1=Slight 2=Moderate 3=Serious 4=Severe			

The solution is very mildly alkaline and would be only slightly corrosive, but the problem of corrosion would be relatively small to steel and most plastics.

Section 3. Safe Usage Data

PROTECTIVE EQUIPMENT TYPES	EYES	Safety glasses. It is generally recognized that contact lenses should not be worn when working with chemicals because they may contribute to the severity of an eye injury.
	RESPIRATORY	Use only with ventilation adequate to keep air concentration below TLV if use generates vapor or mists. If TLV above is exceeded, wear NIOSH approved respirator.
	GLOVES	Rubber
	OTHER	Long-sleeved shirt, trousers, rubber safety shoes, rubber apron
VENTILATION	GENERAL MECHANICAL	Yes
	LOCAL EXHAUST	Preferred to maintain emissions at point of use below the PEL
PRECAUTIONS	HANDLING & STORAGE	Keep in closed container in a dry well-ventilated area away from incompatible materials. Wash thoroughly after handling.
	OTHER	Avoid contact with eyes, skin and clothing.
INCOMPATIBILITY	MATERIALS TO AVOID	Strong oxidizers and acids

Section 4. Emergency Response Data

FIRE	EXTINGUISHING MEDIA	Water
	SPECIAL PROCEDURES	NONE
	UNUSUAL HAZARDS	NONE
EXPOSURE FIRST AID	EYE:	Immediately flush eyes with plenty of water for at least 15 minutes while occasionally lifting upper and lower eye lid. If irritation occurs and persists, obtain medical attention.
	SKIN:	Wash off with running water. If irritation occurs and persists, obtain medical attention.
	INHALATION:	Remove from exposure. If discomfort occurs and persists, obtain medical attention.
	INGESTION:	Rinse mouth and dilute stomach contents with water, or preferably warm milk if available. Call a physician.
SPILLS	STEPS TO BE TAKEN	Small spills may be disposed of by flushing with water. For large spills, contain by soil or other absorbant material.
RQ= 166,000#	WASTE DISPOSAL METHOD	Consult appropriate federal, state and local regulations for disposal of small amounts of copper contaminated absorbant or solution.

Section 5. Physical Hazard Data

FLAMMABILITY	FLASHPOINT	NONE	°F	°C	METHOD USED:
STABILITY	STABLE	<input checked="" type="checkbox"/>	CONDITIONS TO AVOID NONE		
	UNSTABLE	<input type="checkbox"/>	HAZARDOUS DECOMPOSITION PRODUCTS: Thermal decomposition may release CO, CO ₂ or other hazardous gases.		
HAZARDOUS POLYMERIZATION	MAY OCCUR	<input type="checkbox"/>	WILL NOT OCCUR	<input checked="" type="checkbox"/>	AVOID:

Section 6: Health Hazard Data

EFFECTS OF EXPOSURE, SIGNS AND SYMPTOMS:	Human industrial experience has shown no significant inhalation hazard or skin irritation when good personal hygiene practices are followed. Local irritation of the eye may occur from contact with the solution.
PRIMARY ROUTES OF ENTRY	<input type="checkbox"/> INHALATION <input type="checkbox"/> INGESTION <input checked="" type="checkbox"/> SKIN OR EYE CONTACT

Section 7. Physical and Chemical Properties

BOILING PT=	218°F	SPECIFIC GRAVITY:	1.35	VOLATILE COMPONENTS	Water
pH=	9.0	VAPOR DENSITY (AIR=1)	N/A	EVAPORATION RATE	(Water =1) Approx. = 1
SOLUBILITY IN WATER	Complete	WILL DISSOLVE IN	Water		
APPEARANCE	Blue Solution			MATERIAL IS:	LIQUID <input checked="" type="checkbox"/> POWDER
ODOR	Mild - musty				

Section 8. Manufacturer Data

FIRM'S NAME & MAILING ADDRESS: Electrochemical Products Inc. 17000 Lincoln, New Berlin, WI 53151		
Date Prepared or Revised:	Supersedes Those Dated:	EMERGENCY TELEPHONE NO: BUSINESS HOURS: 414-786-9330 Other Times: Chemtrec 800-424-9300 Day or Night
7/15/91	NONE	



UDYLITE®

TECHNICAL DATA SHEET

CUPRAL® Alkaline, non-cyanide copper plating process

CUPRAL™ is a non-cyanide, alkaline process designed to plate thick, adherent, fine-grain copper deposits directly on steel, brass, and zincated aluminum in rack and barrel applications. CUPRAL is excellent as a non-cyanide copper strike and in decorative copper plating applications. It is ideal for masking steel parts prior to heat treating and performs well in continuous strip applications. READ ENTIRE TECHNICAL DATA SHEET BEFORE USING THIS PRODUCT.

HOW TO USE CUPRAL

OPERATING CONDITIONS

Make-up Composition		
Potassium Carbonate	6.0 oz/gal (45 g/L)	
CUPRAL Make-up	33% by volume	
CUPRAL GR	0.1% by volume	
CUPRAL Wetter	0.1% by volume	
	Optimum	Range
Carbonate	2.5 oz/gal (19 g/L)	1.75 to 4.5 oz/gal (13 to 34 g/L)
CUPRAL Maintenance Index	100 Units	75 to 140 Units
CUPRAL Complexor	22% by volume	20 to 28% by volume
CUPRAL GR	0.1% by volume	0.05 to 0.2% by volume
CUPRAL Wetter	0.1% by volume	0.05 to 0.2% by volume
pH at 140 °F (60 °C)	9.8	9.5 to 10.0
Temperature	140 °F (60 °C)	120 to 150 °F (49 to 66 °C)
Cathode Current Density	20 A/ft ²	5 to 35 A/ft ²
Soluble Anode Current Density	10 A/ft ²	5 to 15 A/ft ²
CUPRAL Auxiliary Anode Current Density	75 A/ft ²	50 to 100 A/ft ²
Auxiliary Cathode to CUPRAL Auxiliary		
Anode Area Ratio	20:1	10:1 to 25:1
Plating Cell Current to Auxiliary Cell Current Ratio	10:1	10:1 to 3:1

MAKE-UP

For each 100 gallons (378 L)

Potassium Carbonate	38 lb (17 kg)
CUPRAL Make-Up	33 gallons (125 L)
CUPRAL GR	1 pint (473 mL)
CUPRAL Wetter	1 pint (473 mL)

1. To a clean process tank, add 33 gallons (125 L) of deionized water.

NOTE: Cyanide is a contaminant to this process. If the process tank has been previously used for a copper cyanide process, the tank must be scrubbed and leached with a hot caustic soda solution [4 oz/gal (30 g/L) caustic soda at 120 °F (49 °C)] until no trace of cyanide remains on the tank walls, bottom, or edges. The anode and cathode rails must also be free of cyanide encrustations. The leachate and any other cyanide-bearing materials must be treated and discharged in accordance with effluent regulations pertinent to the point source discharge.

MAKE-UP (Cont.)

2. Add 33 gallons (125 L) **CUPRAL Make-up** and stir until dissolved. DO NOT HAVE THE **CUPRAL AUXILIARY ANODES** IN THE TANK WHEN MAKING UP THE OPERATING BATH.

Check the pH; then slowly and carefully add liquid potassium hydroxide* (caustic potash - 45% solution) until the solution pH reaches 7.5 to 8.0.

*Do not use sodium hydroxide as precipitation may occur.

3. Add 37 pounds (17 kg) potassium carbonate and mix until dissolved.
4. Hang the bagged copper anodes on the anode rails of the main cell (Process Tank). Insert the **CUPRAL Auxiliary Anodes** and steel or copper cathodes into the auxiliary cell, which will be operated on a separate auxiliary rectifier.
5. Bring the operating solution close to its operating volume by adding deionized water. Stir thoroughly.
6. Adjust pH to 9.8 with liquid potassium hydroxide and then adjust the solution level to operating volume.
7. Add 1 pint (473 mL) **CUPRAL GR**.
8. Add 1 pint (473 mL) **CUPRAL Wetter**.
9. Stir well and heat the operating solution to the recommended temperature.
10. Begin circulating solution between the plating cell and the auxiliary plating cell.
11. Process is now ready for plating operations.

OPERATION

CUPRAL Make-up supplies the copper ions, complexor, and electrolyte needed for solution make-up. **CUPRAL Make-up** should only be used for make-up or when recommended by a UDYLITE Technical Representative.

CUPRAL Maintenance supplies the copper ions and stabilizer needed for solution maintenance. **CUPRAL Maintenance** is depleted by electrolysis and dragout. Low concentration of copper metal and electrolyte will produce low cathode efficiency and grainy deposits. High concentration will result in poor adhesion. **CUPRAL Maintenance** must be controlled by daily analysis and additions. The addition of 5 gallons/100 gallons **CUPRAL Make-up** will increase the Maintenance Index by 8 units.

CUPRAL Complexor is used to complex the copper ions, thus, providing adhesion of copper metal to the ferrous base metal. **CUPRAL Complexor** is depleted by electrolysis and dragout. Insufficient **CUPRAL Complexor** will result in poor adhesion. High concentration of **CUPRAL Complexor** will produce low cathode efficiency and grainy deposits.

Carbonate salts are used to buffer the pH, provide conductivity, and promote dissolution of the electrolytic copper anodes. Low concentration of carbonates will cause pH fluctuation and will adversely affect the appearance of the copper deposit. **Potassium carbonate** is recommended for solution make-up. **Potassium Bicarbonate** is recommended for carbonate maintenance.

CUPRAL GR is used to produce a fine-grained, lustrous appearance and makes the copper deposit a suitable substrate for subsequent plating. Insufficient **CUPRAL GR** will result in a grainy, dull appearance which yields unacceptable subsequent plates. Normal consumption rate is 1 quart per 1,300 ampere-hours.

CUPRAL Wetter is used to reduce pitting by lowering the surface tension of the plating solution. If pitting is observed in plated parts, 0.1% (v/v) **CUPRAL Wetter** should be added. If excessive amounts of **CUPRAL Wetter** are required, it may be indicative that the solution is severely contaminated and is in need of a batch carbon treatment.

Operating temperatures below the recommended range may produce grainy and burnt deposits. Temperatures above the recommended range may result in grainy deposits.

Deionized /distilled water must be used for make-up and all volume maintenance additions. **No tap water** is to be added to the **CUPRAL** tank.

If pH adjustment becomes necessary, use liquid potassium hydroxide (45% v/v) to raise the solution pH and glacial acetic acid to lower pH. Variations in pH may result in a grainy deposit and poor adhesion.

Observe **CUPRAL Auxiliary Anodes** once a month for degradation of the surface, causing attack of the substrate.

EQUIPMENT

Tank	Koroseal, PVC lined or approved rubber lined WARNING: Operating temperature over 160 °F (71 °C) may require special high temperature tank lining.
Auxiliary Plating Cell*	See pages 10 and 11 for a detailed description.
Heating Coils	Teflon
Heating Control	Automatic
Ventilation	Required. Solution is classified as B-2 by OSHA.
Plating Cell Rectifier	For full range with less than 5% ripple
Auxiliary Cell Rectifier	0 to 6 Volt rectifier with less than 5% ripple and current capabilities of at least 10 percent of total Plating Cell current
Air Agitation & Spiders*	PVDC
Anodes	Plating Cell: 110 Alloy ETP Copper Auxiliary Cell: CUPRAL Auxiliary Anodes
Auxiliary Cell Cathode	Expanded steel or copper
Copper Anode Bags	
If plating to thickness of:	
a) less than 0.2 Mils	Style 100 7.5 ounce cotton with 48/80 thread count or Style 120 nylon 210 denier with 58/62 thread count
b) greater than 0.2 Mils	Style 7020 napped polypropylene All bags must be desized and washed.
Filtration	Minimum 2 turnovers of solution per hour through a lined or nonmetallic packable plate filter. Filter packing must be composed of cellulose media such as UDYFIN® 984. 1 to 2 pounds of UDYSORB® activated carbon per 1,000 gallons operating solution per week is required.

* Consult an Enthone-OMI Technical Service Representative for design and/or material recommendations.

CONTROL

The following analytical procedures are recommended for use by personnel who have been trained to use laboratory practices which are considered safe and prudent by chemical industry standards. Such practices include suitable personal protective equipment, the use of proper equipment, the use of proper methods of handling all chemicals and proper laboratory procedures.

CAUTION: The following procedures involve the use of potentially hazardous chemicals; manufacturer's material safety data sheet should be consulted and the appropriate safety cautions followed.

DETERMINATION OF MAINTENANCE INDEX

Equipment Needed

- 10 mL pipet
- 50 mL buret
- 50 mL graduated cylinder
- 300 mL Erlenmeyer flask

Reagents Needed

- 0.1 Normality of Sodium Thiosulfate Solution
- 1% Soluble Starch Solution (1 g/100 mL)
- 10% Potassium Iodide Solution (100 g/L)
- Concentrated Hydrochloric Acid - Reagent Grade
- Ammonium Bifluoride - Reagent Grade

Procedure

1. Pipet 10.0 mL of the sample to be analyzed into a 300 mL Erlenmeyer flask.
2. Add 0.5 grams of ammonium bifluoride, 50 mL of deionized water, 15 mL of concentrated hydrochloric acid, 15 mL of potassium iodide solution, and 1 to 2 drops of starch indicator.
3. Titrate with 0.1 Normality of sodium thiosulfate solution to a milky white end point.
4. Maintenance Index = $V \times 6.4$

Where V = mL of sodium thiosulfate solution used in Step 3.

The addition of 5 gallons of CUPRAL Maintenance will increase the Maintenance Index by 8 units/100 gallons.

DETERMINATION OF TOTAL COMPLEXOR

Equipment Needed

- 10 mL pipet
- 50 mL buret
- 300 mL Erlenmeyer flask
- 1000 mL volumetric flask

Reagents Needed

10% Potassium Cyanide Solution - Dissolve 100 grams of reagent grade potassium cyanide in about 700 cc of deionized water. Dilute to 1 liter using a volumetric flask.

CAUTION: Potassium cyanide solution is extremely toxic. Avoid contact with skin or eyes. Do not ingest. Remove and dispose of contaminated clothing. In case of contact or ingestion, seek medical attention immediately!

Magnesium Chloride Solution - Dissolve 20.3 grams \pm 0.1 grams of reagent grade magnesium chloride hexahydrate ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) in about 500 mL deionized water. Dilute to 1 liter using a volumetric flask. Standardize against a known EDTA.

Ammonium Chloride-Ammonium Hydroxide Buffer Solution - Dissolve 50 grams of reagent ammonium chloride in 400 mL of deionized water and add 400 mL of reagent grade (28%) ammonium hydroxide. Dilute to 1 liter using a volumetric flask.

Eriochrome Black T Indicator - To 100 grams of reagent grade sodium chloride add 0.20 grams of Eriochrome Black T dye. Mix thoroughly and store in a closed amber bottle.

DETERMINATION OF TOTAL COMPLEXOR

Procedure

1. Pipet 10 mL of the sample to be analyzed into a 300 mL Erlenmeyer flask.
2. Using a graduated cylinder, add 15 mL of ammonium hydroxide buffer and 15 mL of deionized water.

DETERMINATION OF TOTAL COMPLEXOR (Cont.)

3. Slowly add 10% potassium cyanide until the blue color is discharged and the solution becomes colorless; then add 1 mL more of the cyanide solution. Do not add an excess. Approximately 5 to 10 mL will be necessary.
4. Add a small amount (0.1) of the solid Eriochrome Black T indicator until the solution develops a blue color. Too intense a color will make the end point hard to see.
5. Titrate with 0.1 Molar magnesium chloride solution until the solution turns from blue to purple. (If the end point is passed, the solution turns a pink color. The true end point is the change from blue to purple.)
6. Properly discard the solution as a cyanide-containing waste.
7. % by volume total **CUPRAL Complexor** = $V \times M \times 14.3$
V is the Volume of magnesium chloride.
M is the Molarity of magnesium chloride.

DETERMINATION OF CARBONATE

Equipment Needed

- 5 mL pipet
- 25 mL graduated cylinder
- Chittick Gasometric Apparatus - see page 12 for design of this apparatus.
(VWR Catalog #19750-00 Carbon Dioxide Apparatus)

Reagents Needed

6 Normal Sulfuric Acid (100 mL/500 deionized water)

Procedure

1. Pipet 5.0 mL of the sample to be analyzed into the gas evolution flask A (see diagram on page 6).
2. Connect flask A to the Chittick Gasometric Apparatus, allow to stand for several minutes to attain equilibrium, open stopcock C and adjust the liquid level in the burette D to 15.0 mL above 0 by raising leveling bulb E.
3. Close stopcock C and slowly add 15 mL of 6 Normality of sulfuric acid solution from burette F to flask A while slowly lowering the leveling bulb E, keeping the liquid level in E slightly lower than the liquid level in burette D.
4. Vigorously swirl the contents in flask A to evolve all the CO₂ and allow to stand for several minutes to attain equilibrium.
5. Raise the leveling bulb E until the liquid level in it is at the same level as the liquid level in burette D. Read the volume of gas evolved in mL in burette D which is the reading of the liquid level in D.
6. oz/gal Carbonate = $V \times 0.070$
V is the Volume of CO₂ gas evolved as measured in Step 5.

NOTE: Accuracy of the method is sufficient without using a temperature or atmospheric pressure correction when measuring the gas volume in Step 5.

CUPRAL CARBONATE EQUIVALENTS

CARBONATE ADDITION	POTASSIUM CARBONATE (43.5% CO ₃)	POTASSIUM BICARBONATE (60% CO ₃)
0.25 oz/gal	0.57 oz/gal	0.42 oz/gal
0.5 oz/gal	1.1 oz/gal	0.83 oz/gal
1 oz/gal	2.3 oz/gal	1.7 oz/gal
2 oz/gal	4.6 oz/gal	3.4 oz/gal
2.61 oz/gal (standard)	6 oz/gal	4.35 oz/gal
3 oz/gal	6.9 oz/gal	5 oz/gal
3.5 oz/gal	8 oz/gal	5.8 oz/gal
4 oz/gal	9.2 oz/gal	6.7 oz/gal

DETERMINATION OF WETTER IN CUPRAL

Prior to using the stalagmometer tube, thoroughly clean it by soaking it for several hours in chromic acid solution followed by thorough and careful rinsing. This cleaning operation need not be carried out for each use of the stalagmometer. After the primary cleansing of the stalagmometer, a thorough rinsing with distilled water is sufficient. However, prior to determination of the surface tension, the stalagmometer must be rinsed with a portion of the solution to be tested.

Do not touch the polished dropping surface with the fingers since it must be clear and free of grease.

Mount the stalagmometer solidly in a vertical position on a ring stand. Make sure there is no vibration.

Procedure

1. Adjust the temperature of all solutions to be tested to 68 °F (25 ± 1 °C).
2. Rinse the stalagmometer with the test solution.
3. Fill the stalagmometer with **CUPRAL** test solution. Adjust the solution level to the upper zero. Wipe the base of the stalagmometer.
4. Now allow the solution to drain out of the stalagmometer, counting the number of drops delivered as the solution flows between the upper zero level to the lower zero level of the instrument.
5. Record the number of drops.
6. To insure that the results are consistent, Steps 3 to 5 should be repeated three more times. Calculate the average of all the attempts to the nearest drop.
7. Rinse the stalagmometer several times using distilled water.
8. Repeat Steps 1 to 6 substituting distilled water to calibrate the stalagmometer. This is done infrequently in order to calibrate the instrument.
9. Using a hydrometer, determine the specific gravity of the **CUPRAL** solution. Use this value for the density (Dtc) of the solution.

DETERMINATION OF WETTER IN CUPRAL (Cont.)

Calculation

Calculate the surface tension (ST) in dynes/cm using the following formula:

$$ST = \frac{ST_w \times D_{tc} \times N_w}{D_w \times N_{tc}}$$

Where:

ST = Surface tension of **CUPRAL** chromium solution
 ST_w = Surface tension of distilled water
 D_{tc} = Density of **CUPRAL** solution
 D_w = Density of distilled water
 N_w = Number of drops of distilled water
 N_{tc} = Number of drops of **CUPRAL**

<u>Standard Solution</u>	<u>Surface Tension</u>	<u>Density</u>
Distilled Water	72	1.0

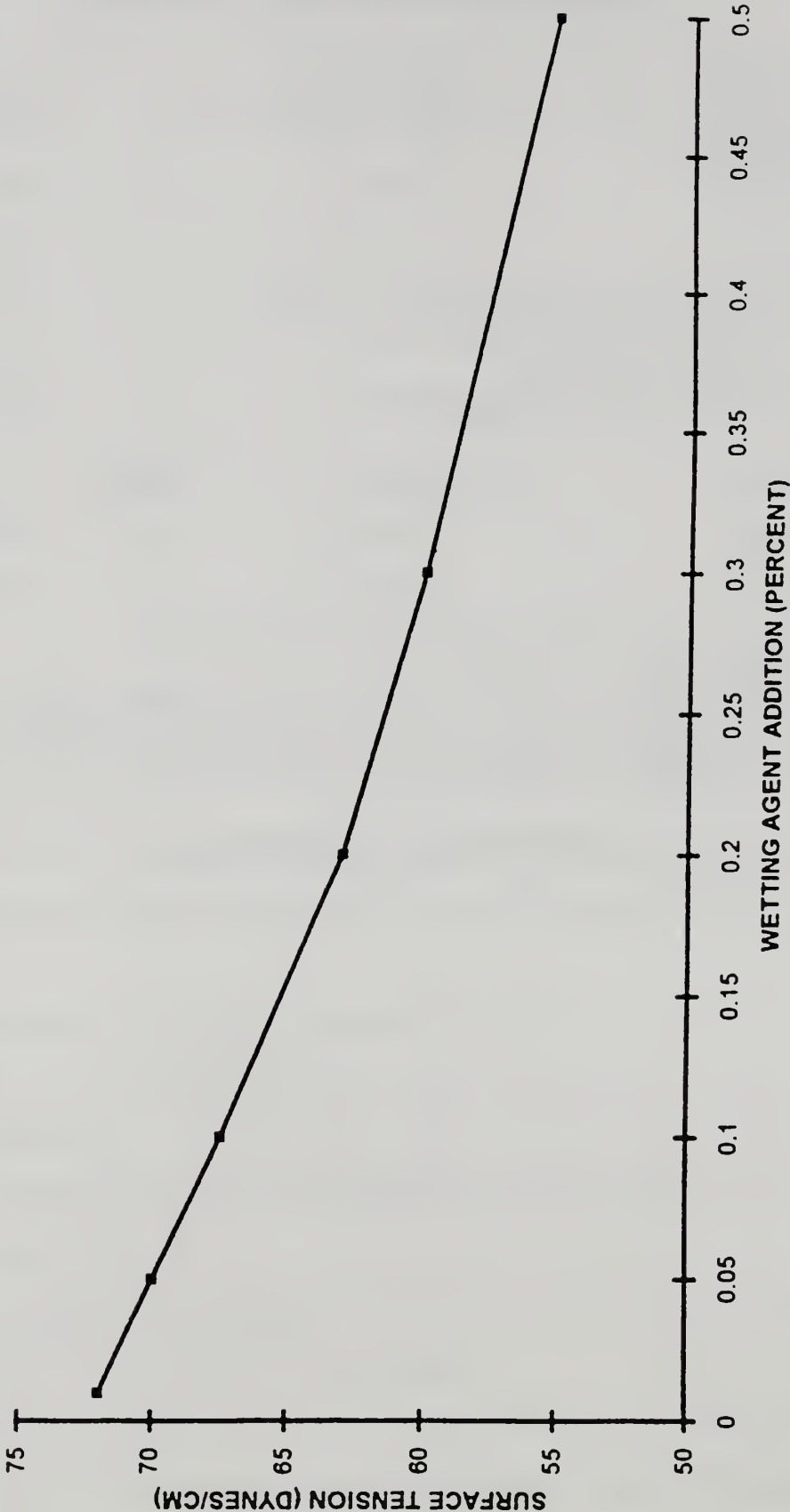
EXAMPLE:

Drops CUPRAL solution	74
Density CUPRAL solution	1.310
Drops of distilled water	36

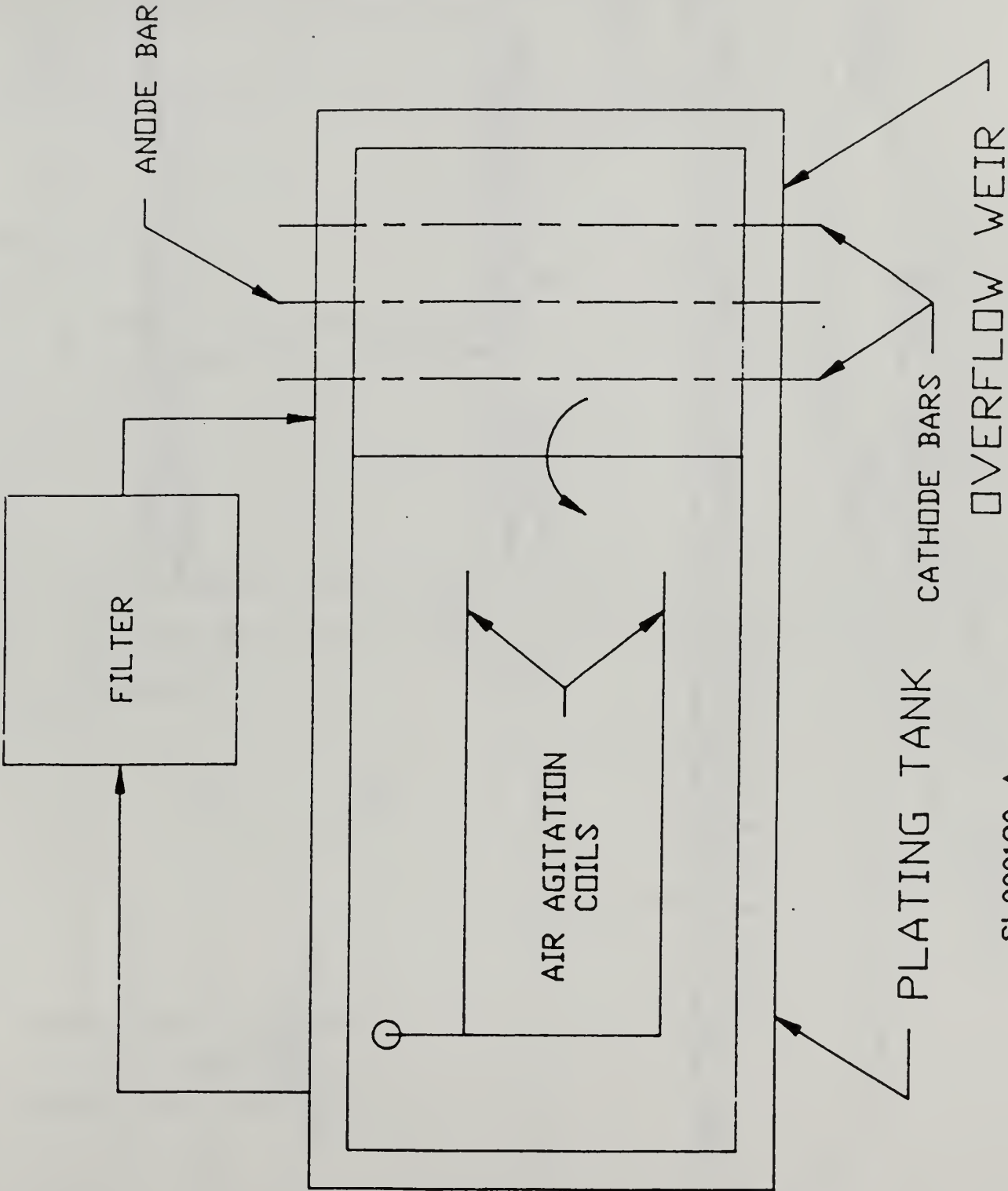
$$ST = \frac{72 \times 1.310 \times 36}{1.0 \times 74} = 45.9 \text{ dynes/cm}$$

Determine concentration of **CUPRAL Wetter** from the following chart.

SURFACE TENSION EFFECT OF CUPRAL WETTING AGENT

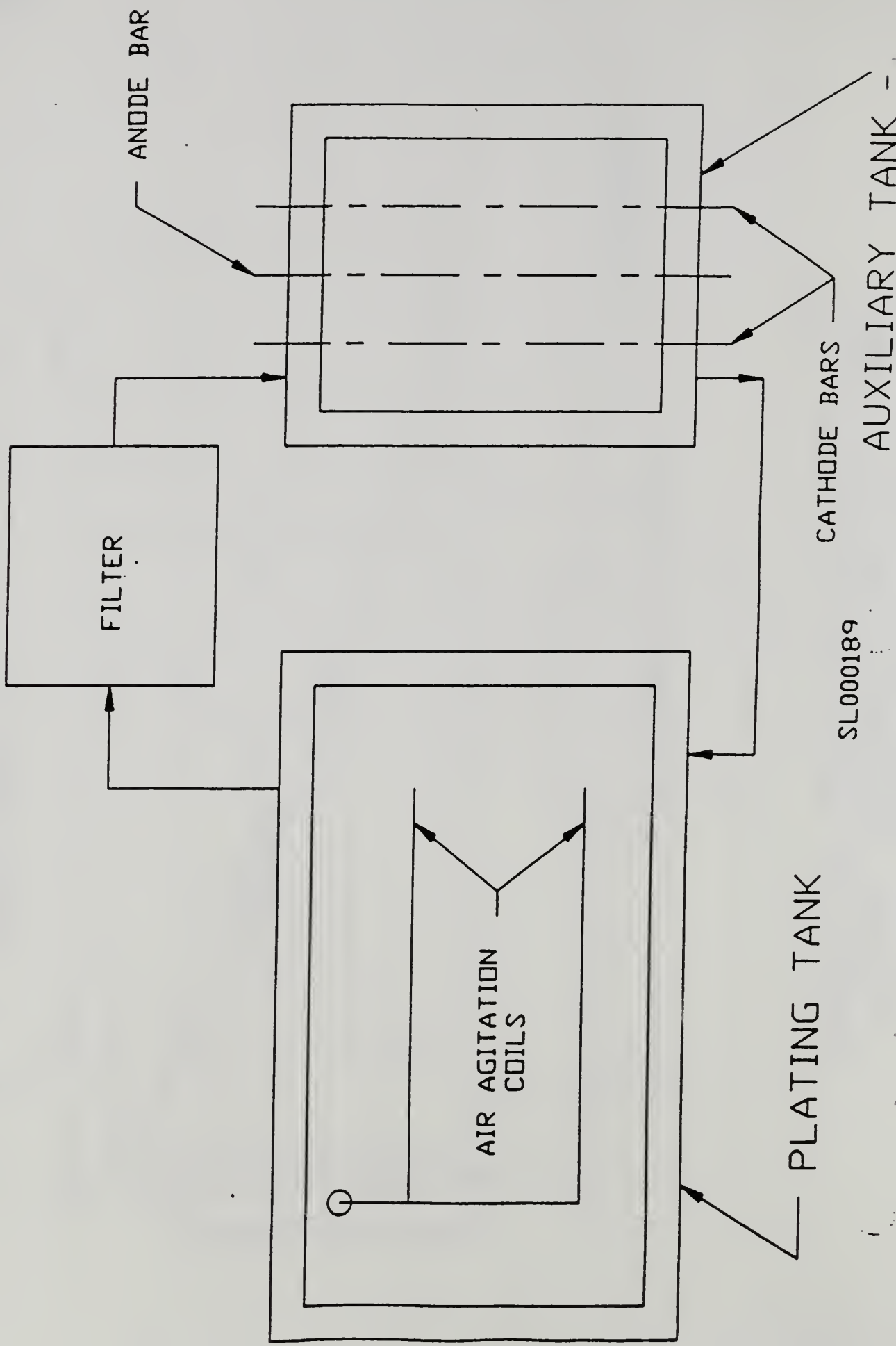


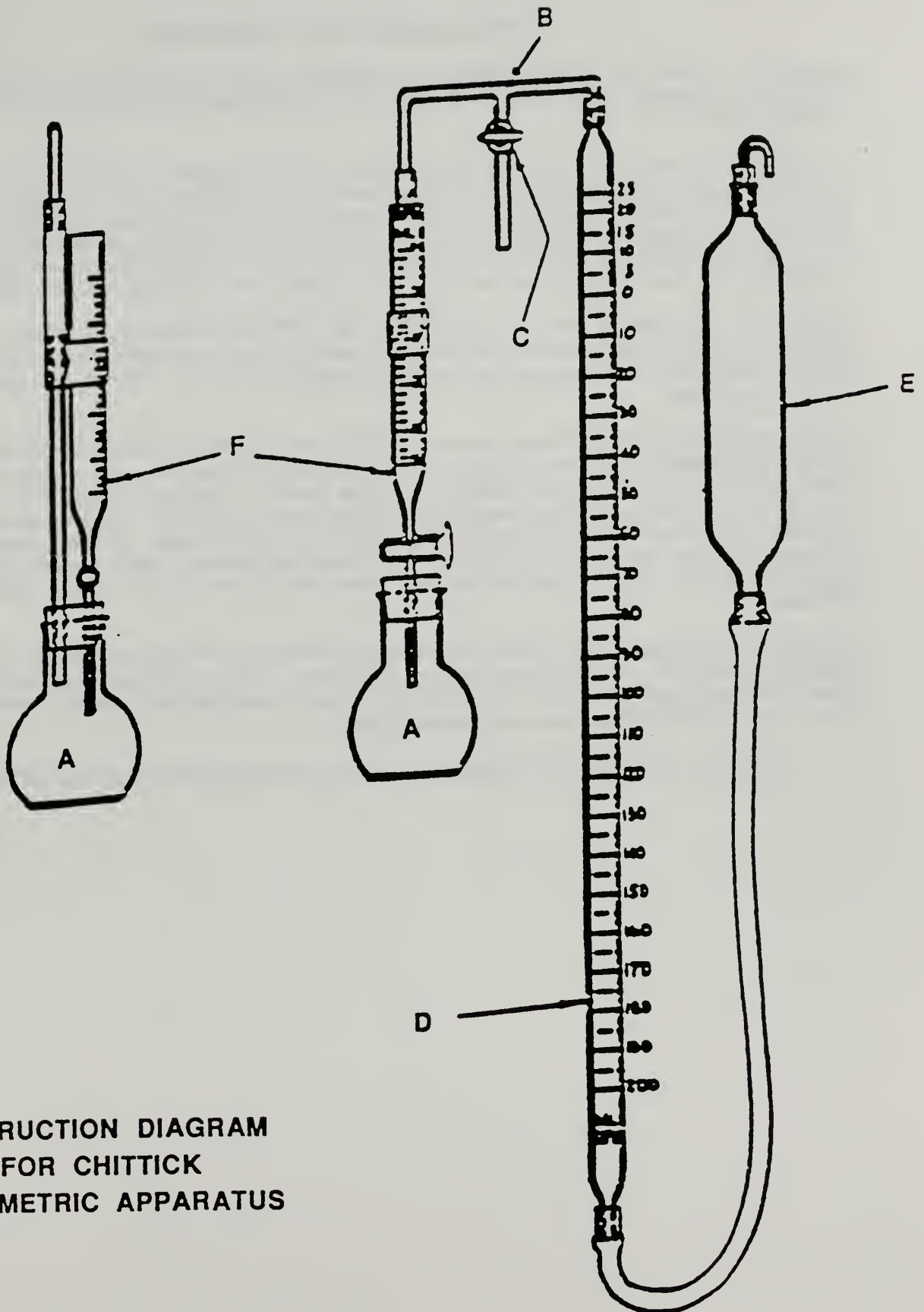
CUPRAL SCHEMATIC DESIGN USING



SL000190-A

CUPRAL SCHEMATIC DESIGN USING
SEPARATE AUXILIARY TANK





INSTRUCTION DIAGRAM
FOR CHITTICK
GASOMETRIC APPARATUS

PRECAUTIONARY INFORMATION

DANGER! CUPRAL GR MAY CAUSE SEVERE BURNS. CUPRAL MAKE-UP, CUPRAL Wetter, CUPRAL Complexor, CUPRAL Maintenance Index OR THE OPERATING SOLUTION MAY CAUSE IRRITATION, EYE DAMAGE.

HAZARDS: CUPRAL GR may cause severe burns to skin and eyes. Inhalation may cause respiratory tract irritation. Ingestion may cause severe burns. CUPRAL MAKE-UP, CUPRAL Wetter, CUPRAL Complexor, CUPRAL Maintenance Index or the operating solution may cause irritation to skin and eyes, possible eye damage. Inhalation of vapors may cause irritation to the respiratory tract. Ingestion may cause severe irritation to the gastro-intestinal tract. CONSULT SUPPLIERS MSDS's FOR ADDITIONAL INFORMATION. Do not get in eyes, on skin, or on clothing. Do not inhale or take internally.

FIRST AID: In case of contact of CUPRAL GR, CUPRAL MAKE-UP, CUPRAL Wetter, CUPRAL Complexor, CUPRAL Maintenance Index or the operating solution with skin or eyes, flush with plenty of clean, cool water for 15 minutes. Get medical attention for eyes. Remove and wash contaminated clothing and shoes.

HANDLING INFORMATION: When handling CUPRAL GR CUPRAL MAKE-UP, CUPRAL Wetter, CUPRAL Complexor, CUPRAL Maintenance Index or the operating solution, wear protective clothing, chemical safety goggles, respirator, face shield and rubber gloves. Exhaust ventilation is recommended to remove vapors that may be generated during Make-Up and Operation. When preparing or adding to solutions, always add CUPRAL GR CUPRAL MAKE-UP, CUPRAL Wetter, CUPRAL Complexor, CUPRAL Maintenance Index slowly and cautiously. Avoid breathing vapors. Avoid contact with acids, oxidizing agents, alkalies or any other foreign material. Do not get in eyes, on skin, or on clothing. Do not take internally.

CONTAINER INFORMATION: Keep containers tightly closed. Keep closed when not in use to avoid moisture absorption. Store indoors in a cool, dry area. Loosen closure cautiously when opening. Wash thoroughly after handling. Do not reuse containers, wash before disposal. Improper disposal or reuse of this container may be dangerous and illegal.

REFER TO MSDS FOR FURTHER SAFETY AND HANDLING INFORMATION.

Issued: 11/05/93
Spsds: 02/03/92

MATERIAL SAFETY DATA SHEETS

For more detailed information on the toxicological properties of the products described herein, reference can be made to the Material Safety Data Sheet (MSDS) for each product. If you do not have the proper MSDS, it can be requested from: Enthone-OMI Inc., attention: Regulatory Affairs Department, P.O. Box 1900, New Haven, CT 06508. For emergency assistance call CHEMTREC (800) 424-9300.

WARRANTY AND DISCLAIMER

The information presented herein is to the best of our knowledge true and accurate and all recommendations and suggestions appearing in this bulletin covering the use of our products are based upon information believed to be reliable. However, since the conditions of use are beyond our control, this information is given on the express condition and agreement that Enthone-OMI Inc. will not be liable to any person in contract, tort (including negligence), strict liability or otherwise for any claims, damages or losses whatsoever. Nothing herein shall be deemed a recommendation to use any product or process in violation of any existing patent rights and no warranties, expressed or implied, are made regarding the information, product, processes, recommendations, description and safety notations contained herein. The above includes proprietary information of Enthone-OMI Inc. and is furnished to you for your use solely on products or processes supplied by us to you.

Enthone-OMI (Canada) MATERIAL SAFETY DATA SHEETS

For more detailed information on the toxicological properties of the products described herein, reference can be made to the Material Safety Data Sheet (MSDS) for each product. If you do not have a current MSDS, it can be requested from the W.H.M.I.S. co-ordinator, Enthone-OMI (Canada) Inc., 58 Adesso Drive, Concord, Ontario, L4K-3C6. For emergency assistance regarding accidents with this product resulting in container rupture, spills, poisoning, bodily injury or threats to health call: CHEMTREC (800) 424-9300.

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APPENDIX B E-Brite 30/30 Bath Analysis Techniques



Electrochemical Products, inc.

17000 Lincoln Avenue New Berlin, WI 53151-2781 (414) 786-9330 FAX (414) 786-9403

TECHNICAL DATA

Determination of E-Brite 30/31 in E-Brite 30/30

1. Pipet 10 ml. of the E-Brite 30/30 plating solution and place it in a 600 ml. beaker. Add 50 ml. of D.I. water to this sample.
2. Under magnetic stirring and using a pH meter adjust pH to just below 4.0 with concentrated Hydrochloric Acid.
3. Add the 1.0N Sodium Hydroxide from a burette and raise the pH of the solution to 4.6.
4. Zero the burette with 1.0N Sodium Hydroxide and titrate the solution using a standardized pH meter, to pH 11.2.
5. Record the number of mls. of 1.0N NaOH used to titrate.

$$\text{E-Brite 30/31, \% by vol.} = \text{mls. 1.0N NaOH} \times 3.8$$

Minimum amount of E-Brite 30/31 electrolyte required to operate E-Brite 30/30 bath corresponding to the amount of copper metal contained in it.

COPPER METAL (oz/gal.)

E-Brite 30/31, \% by volume

1.0	40%
1.1	44%
1.2	48%
1.3	52%
1.4	56%
1.5	60%
1.6	64%
1.7	68%
1.8	72%
1.9	76%
2.0	80%
2.1	84%
2.2	88%
2.3	92%
2.4	96%
2.5	100%

Example:

If the copper metal is 1.7 oz/gal., your E-Brite 30/30 bath should have a minimum of 68% by volume E-Brite 30/31. This is your theoretical value.

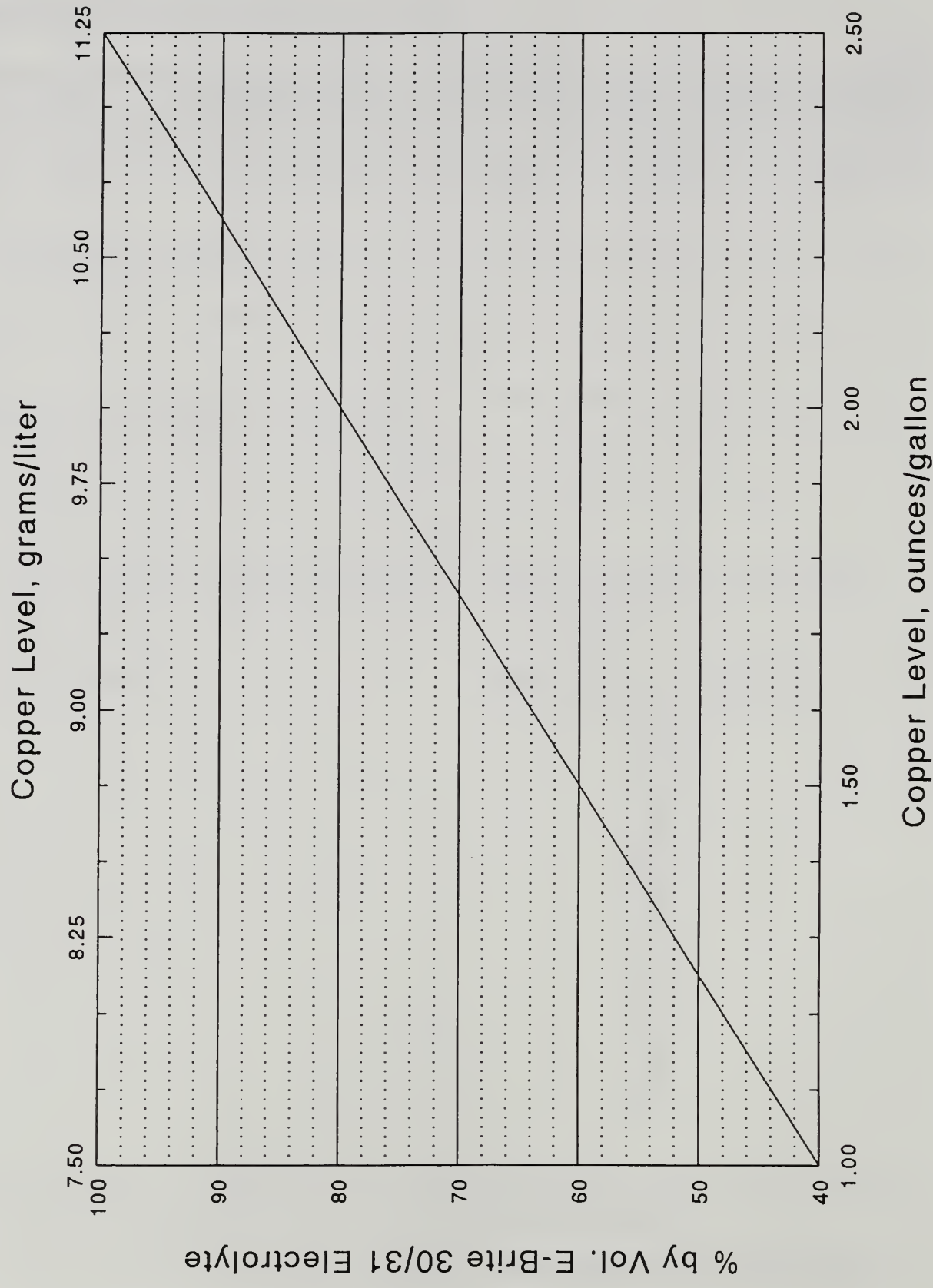
Let's say you titrated 16.8 mls. of 1.0N NaOH. Your actual value E-Brite 30/31 is 64%.

To determine amount of 30/31 needed, take your theoretical value minus your actual value equals the % 30/31 needed to be added. For this titration $68 - 64\% = 4\%$ by volume E-Brite 30/31 needed to be added to the bath.

NOTE:

Presence of metallic and organic impurities, which are normal in E-Brite 30/30 production plating, will require additional E-Brite 30/31 over and beyond that needed for the copper in solution. Such additions of E-Brite 30/31 are to be determined based on Hull cell plating and testing for plate adhesion.

Figure 8 E-Brite 30/31 Electrolyte
Min. % by Vol. Req'd for Copper Level



**Determination of Copper Content and Other Metallic Impurities in
the E-Brite 30/30 Bath by ICP-AES**

Prepare 100 mls of a multielement standard for copper (Cu), iron (Fe), nickel (Ni), and zinc (Zn). Pipet the quantities of 1000 ppm single element standards required in Table I into a 100 ml volumetric flask. Dilute to 100 ml mark with 18 megaohm deionized water. Label the standard with its concentrations, and expiration date.

Table I 100 ml Multielement Standard for E-Brite 30/30 Bath

Multielement Standard Concentration	Amount of 1000 ppm Single Element Standard Required
20 ppm Cu	2.0 mls
2 ppm Fe	0.2 mls
2 ppm Ni	0.2 mls
2 ppm Zn	0.2 mls

Pipet 100 uls of the E-Brite 30/30 bath into a 100 ml volumetric flask. The sample of the bath should be at room temperature. Dilute to the 100 ml mark with 18 megaohm deionized water. Label the sample with its identity and date.

Utilize the wavelengths in Table II for analysis of the sample on the Perkin Elmer ICP (Model 6500) sequential spectrometer.

Table II Element Wavelengths for E-Brite 30/30 Bath ICP Analysis

Element	Wavelength, nm.
Copper	324.754
Iron	259.940
Zinc	206.200
Nickel	231.604

Convert the obtained results into the desired units utilizing the following formulas.

ICP result in ppms or mg/l $\times .1334$ = oz/gal

ICP result in ppms or mg/l $\times 1$ = g/l

APPENDIX C Electrical Testing Report

To: Martha Jowett

6/14/94 4240:WJ:94-050

From: Bill Jundzil

Subject: G6 Flexleads - Process Change
Toxic Chemical Elimination Program

SUMMARY:

In order to reduce toxic chemical use, a new process of plating G6 flexleads was tried. As part of the testing of this new plating process, measurements were made to determine if the current carrying capability of the flexleads was degraded.

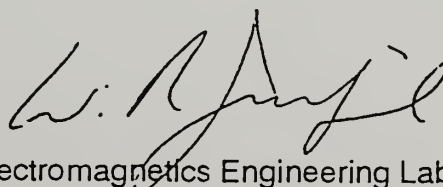
No degradation resulted from the new process. Both the standard process and the new process flexleads performed the same. The average fuse current for the standard process flexleads was 4.1 amps. The new process flexleads had an average fuse current of 4.0 amps.

TEST PROCEDURE:

Three groups of G6 flexleads, part no. 69387, were sent to the EM Lab for analysis. Group 1 flexleads, control group, were plated using the standard method copper cyanide solution. Groups 2 and 3 were plated in the new solution, E-Brite 30/30. Group 3 flexleads received extra stripping time in the chromic acid solution. Groups 2 and 3 were fabricated under OCN 6666.

The test consisted of immersing 3 flexleads from the same group in a petri dish full of gyro bromo fluid. (Due to degradation of the bromo from the heat, the bromo was replaced after each test.) A 800 Hz current, starting at 1 amp, was applied for 10 minutes. If no fusing of the leads occurred, the current was increased by 1/4 amp and held for another 10 minutes. This was repeated up to the 3 amp level (past history shows leads fusing at 2.5 amps and above). The current was then slowly increased until the flexleads fused. This test was repeated, 3 new flexleads per test, until all leads were measured. The fuse current for each lead is shown below.

FUSE CURRENT (AMP-TRMS)				
CuCn Group 1	E-Brite Group 2	E-Brite (restrip) Group 3	4/13/93 Data	Past History
3.6	3.5	3.9	3.8	>2.5
3.7	3.7	4	4.2	
3.9	3.7	4.1	4.3	
4.2	3.8	4.2	4.4	
4.5	3.9	4.5	4.4	
-----	4	-----	4.4	
	4.1			
	4.2			



Electromagnetics Engineering Lab

cc: W. Walker

